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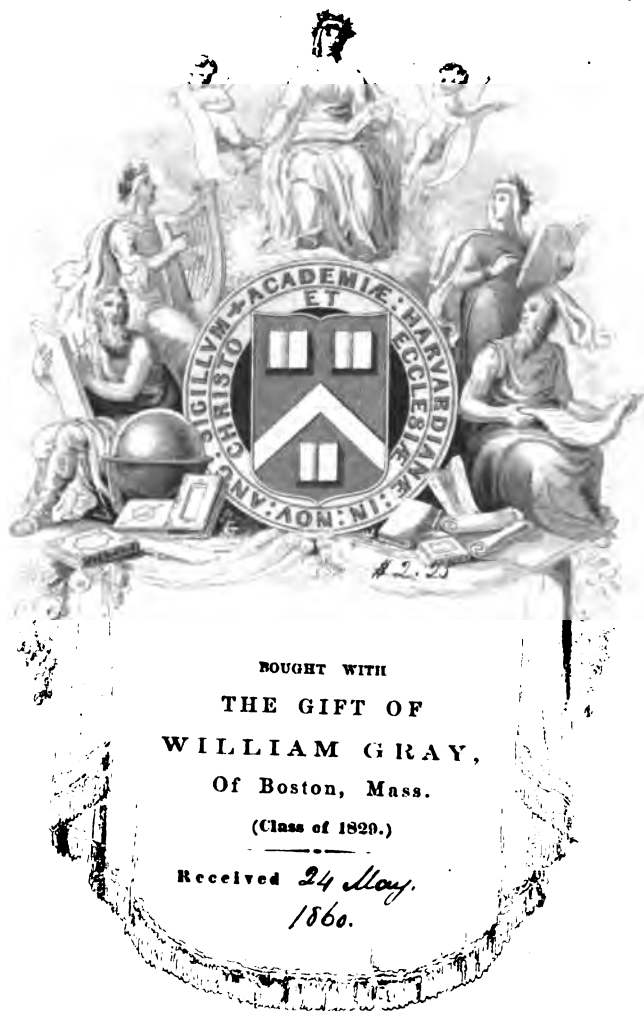
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THE
USE OF THE BLOWPIPE.

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THE
USE OF THE BLOWPIPE
IN THE
QUALITATIVE AND QUANTITATIVE EXAMINATION
OF
MINERALS, ORES, FURNACE PRODUCTS,
AND OTHER METALLIC COMBINATIONS.

BY
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INFLUENCE OF CHEMISTRY; CHEMISTRY, THEORETICAL, PRACTICAL, AND
ANALYTICAL, AS APPLIED AND RELATING TO THE ARTS
AND MANUFACTURES, ETC. ETC. ETC.

Illustrated by Numerous Diagrams.

"Segnius irritant animos demissa per aurem,
Quam quæ sunt oculis subjecta fidelibus."—HORACE.

THIRD EDITION, REVISED AND FURTHER ENLARGED.

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P R E F A C E

TO THE THIRD EDITION.

THE interest excited in this country and America by this work, and the high encomiums that have been bestowed upon it by distinguished scientific men at home and abroad, all concurring in the opinion that the information and appliances it contains are most essential to the Chemist, Geognogist, Geologist, Mineralogist, Metallurgist and Agriculturist, have induced the call for a Third Edition,—a proof of the increasing aptitude for this species of research.

In preparing the present Edition, the English Author has endeavored to render it deserving of a continuance of the favor with which the previous ones were received.

For that purpose, the subject of each chapter has been carefully reconsidered, and, in several instances, the matter almost entirely rewritten ; many new drawings of apparatus have also been introduced, so that the results of the numerous valuable researches in this department of Chemistry which have been added to science within the last few years, will be found incorporated in their proper places, with as much detail as was due to their respective importance. When the work was first issued, Dr. MUSPRATT felt satisfied that its technical nature would preclude an

extensive circulation, but that eventually it would come into general use. At the present time it is a work of reference in every laboratory in Europe and America ; moreover, numbers of copies have found their way to Australia.

The truthfulness of its results are daily witnessed, for it enables the mere beginner to discover the presence of Antimony, Arsenic, Bismuth, Chromium, Cobalt, Copper, Lead, Magnesia, Manganese, Mercury, Selenium, Silver, Soda, Strontia, Sulphur, and Zinc, and a skilful operator can prove *infallibly* the presence of a still greater number of substances.

Dr. MUSPRATT takes this opportunity of expressing his best thanks to his talented student, Mr. E. E. HOLDEN, for the valuable assistance he has rendered him in preparing this work for the press.

*College of Chemistry, Liverpool,
October, 1854.*

CONTENTS.

SECTION I.

DESCRIPTION OF THE BLOWPIPE, MANIPULATING APPARATUS, ET CETERA.

	Page
I. THE BLOWPIPE	1
II. THE COMBUSTIBLE MATERIAL	7
III. THE BLOWING, ET CETERA, AND DESCRIPTION OF THE FLAME ...	10
The Oxidation Flame	11
The Reduction Flame	12
IV. THE SUPPORTS :—	
1. Charcoal	13
2. Platinum Wire	14
3. Platinum Foil	14
4. Glass Tubes	15
5. Glass Flasks	15
6. Platinum Spoons	15
7. Clay Basins	16
8. Clay Crucibles	17
9. Soda-Paper... ..	19
10. A Mixture of seven parts of Charcoal, with one part of Clay ...	19
V. INSTRUMENTS, ET CETERA, REQUISITE IN ANALYSIS WITH THE BLOWPIPE :—	
1. A Balance	20
2. A set of Weights	22
3. A Microscope	22
4. Forceps	22
5. A Mortar and Pestle of Agate, or Calcedony	23

	Page
6. A Hammer	24
7. An Anvil	24
8. Charcoal Borers	25
9. Charcoal Saw	26
10. Charcoal Holder	26
11. Platinum Wire and Foil	27
12. A Capsule of Brass Foil	28
13. A small Scissors	28
14. A Knife	28
15. Files	28
16. An Iron Instrument for the formation of Cupels	29
17. An Ivory Spoon	30
18. A small Brush	30
19. A Lead Sieve	30
20. A Steel Magnet	30
21. A Lead Measure	30
22. A small Wooden Cylinder	31
23. A Polished Iron Spatula	31
24. Test Tubes	31
25. A Glass Pipette	32
26. An Edulcorator	32
27. Watch Glasses	33
28. Glazed Porcelain Basins	34
39. A Cylindrical Bottle, of sheet iron, for Lamp-Oil	34

VI. BLOWPIPE REAGENTS :—

Reagents in the Humid way.

I. GENERAL REAGENTS.

a. Reagents used as Simple Solvents.

1. Distilled Water	35
2. Alcohol	35
3. Ether	36

b. Reagents principally employed as Chemical Solvents.

1. Hydrochloric Acid	36
2. Nitric Acid	37
3. Nitrohydrochloric Acid, Aqua Regia	37
4. Acetic Acid	37
5. Chloride of Ammonium	38

c. Reagents used to separate, or otherwise characterize Groups of Substances.

1. Reagent Papers	
<i>a.</i> Blue Litmus Paper	38
<i>b.</i> Reddened Litmus Paper	38
<i>c.</i> Brazil Wood Paper	38
<i>d.</i> Turmeric Paper	39

CONTENTS.

ix

	Page
2. Sulphuric Acid	39
3. Sulphide of Hydrogen	39
4. Sulphide of Ammonium	41
5. Solution of Potassa	41
6. Ammonia	41
7. Carbonate of Ammonia	42
8. Chloride of Barium	42
9. Nitrate of Baryta	42
10. Chloride of Calcium	43
11. Nitrate of Silver	43
12. Sesquioxide of Iron	43

II. SPECIAL REAGENTS.

a. *Reagents used for the detection of Bases.*

1. Sulphate of Potassa	44
2. Chromate of Potassa	44
3. Cyanide of Potassium	44
4. Ferricyanide of Potassium	44
5. Ferrocyanide of Potassium	45
6. Sulphocyanide of Potassium	45
7. Phosphate of Soda	45
8. Oxalate of Ammonia	45
9. Protochloride of Tin	45
10. Bichloride of Platinum	45
11. Trichloride of Gold	46
12. Zinc	46
13. Copper	46
14. Iron	46

b. *Special Reagents employed for determining the presence of Acids.*

1. Acetate of Potassa	47
2. Hydrate of Lime	47
3. Sulphate of Lime	47
4. Sulphate of Magnesia	47
5. Chloride of Magnesium	48
6. Sulphate of Iron	48
7. Neutral Acetate of Lead	48
8. Sulphate of Copper	48
9. Subnitrate of Mercury	49
10. Oxide of Mercury	49
11. Chloride of Mercury	49
12. Sulphurous Acid	49
13. Chlorine	49
14. Sulphindigotic Acid	50
15. Starch Paste	50

<i>Reagents in the Dry Way.</i>		Page
FLUXES AND BLOWPIPE REAGENTS.		
1. Carbonate of Soda	...	50
2. Biborate of Soda	...	51
3. Phosphate of Soda and Ammonia	...	52
4. Nitrate of Potassa	...	52
5. Bisulphate of Potassa	...	53
6. Vitriified Boracic Acid	...	53
7. Nitrate of Cobalt in Solution	...	53
8. Oxalate of Nickel	...	54
9. Black Oxide of Copper	...	55
10. Silica, or Silicic Acid	...	55
11. Fluor Spar	...	55
12. Sulphate of Lime	...	55
13. Bone Ashes	...	56
14. Proof Lead	...	56
15. Chloride of Sodium	...	57
16. Charcoal Powder	...	57
17. Graphite	...	57
18. Tin	...	58
19. Silver Wire	...	58
20. Tartaric Acid	...	58

SECTION II.

QUALITATIVE ANALYSIS WITH THE BLOWPIPE.

GENERAL RULES FOR QUALITATIVE BLOWPIPE EXAMINATIONS:—

General Rules by which Ingredients of Minerals, <i>et cetera</i> , may for the most part be detected, when heated alone, or with reagents in the Blowpipe Flame	...	62
1. Treatment of the Substance with Borax	...	67
2. Treatment of the Substance with Microcosmic Salt	...	70
3. Treatment of the Substance with Carbonate of Soda	...	71
<i>a.</i> Fusibility of a Substance with Carbonate of Soda	...	71
<i>b.</i> Reduction of Metallic Oxides by means of Carbonate of Soda	...	72
General Rules for Qualitative Blowpipe Analysis, by means of which, partly with the assistance of the moist way, the single ingredients of compound bodies may be detected	...	76

CONTENTS.

xi

	Page
1. Smelting of the Substance with Borax and Carbonate of Soda	77
2. Method of treating with Hydrochloric Acid the Substance melted with Carbonate of Soda and Borax	79
3. Smelting of the Substance with Nitrate or Bisulphate of Potassa	80
4. Solution in Water of the Substance fused with Saltpetre or Bisulphate of Potassa	83
5. Precipitation—by crystallized or pulverulent reagents—of single Constituents dissolved in Fluids	84
6. Decantation, Filtration, and Edulcoration	84
7. Desiccation of the Filtered Residues and Precipitates ..	85
Reactions before the Blowpipe of different Oxides, <i>et cetera</i>	86
Examination in the Borax Bead	87
Examination in the Microcosmic Salt Bead	87

BLOWPIPE TABLES:—

Comportment of the Alkalies— <i>Potassa, Soda, Lithia, and Ammonia</i> —alone, and with reagents in the Blowpipe Flame	88, 89
Comportment of the Earths— <i>Baryta, Strontia, Lime, Magnesia, Alumina, Glucina, Yttria, Terbia, Erbica, Zirconia, Thoria, and Silica</i>	88, 89
Comportment of Metallic Oxides— <i>Sesquioxide of Cerium, Oxide of Lanthanum, Oxide of Didymium, Sesquioxide of Manganese, Sesquioxide of Iron, Oxide of Cobalt, Oxide of Nickel, Oxide of Zinc, Oxide of Cadmium, Oxide of Lead, Peroxide of Bismuth, Sesquioxide of Uranium, Oxide of Copper, Oxide of Silver, Oxide of Mercury, Binoxide of Platinum, Binoxide of Palladium, Sesquioxide of Rhodium, Binoxide of Iridium, Oxide of Ruthenium, Peroxide of Gold, Binoxide of Tin, Peroxide of Antimony, and Sesquioxide of Chromium</i>	90-96
Comportment of Acids— <i>Tellurous Acid, Molybdic Acid, Niobic Acid, Pelopie Acid, Tungstic Acid, Vanadic Acid, Tantalie or Columbic Acid, and Titanic Acid</i>	98-99

DESCRIPTION of the PHENOMENA presented by the Sublimates of some Metals, when melted or heated strongly upon Charcoal before the Blowpipe Flame:—

1. Selenium	98
2. Tellurium	99
3. Arsenic	100
4. Antimony	100
5. Bismuth	100
6. Lead	100
7. Cadmium	100
8. Zinc	101
9. Tin	101
10. Silver	101
11. Iodine, Bromine, Chlorine, and Sulphur	101

QUALITATIVE EXAMINATION of Minerals, Ores, and the Products of Metallurgical Operations, for Metallic and Non-Metallic Bodies, before the Blowpipe :—

a. Examination of the Metallic Oxides which form Alkalies and Earths :—

1. Potassa	102
2. Soda	104
3. Lithia	105
4. Ammonia	107
5. Baryta	107
6. Strontia	110
7. Lime	113
8. Magnesia	125
9. Alumina	129
10. Glucina	138
11. Ytria	140
12. Zirconia	149
13. Thorina	152

β. Examination of Metals and their Oxides :—

1. Cerium, Lanthanum, and Didymium	154
2. Manganese	160
3. Zinc	163
4. Cobalt	166
5. Nickel	168
6. Iron	173
7. Cadmium	181
8. Lead	182
9. Bismuth	185
10. Uranium	188
11. Copper	190
12. Silver	193
13. Mercury	201
14. Platinum, Palladium, Rhodium, Iridium, and Osmium	203
15. Gold	204
16. Tin	207
17. Antimony	209
18. Molybdenum	213
19. Tungsten	215
20. Vanadium	217
21. Tantalum, Niobium, and Pelopium	219
22. Titanium	224
23. Chromium	227
24. Tellurium	229

CONTENTS.

xiii

	Page
25. Arsenic	230
26. Selenium	237
γ. Examination for Nonmetallic Bodies and Acids:—	
1. Oxygen and Hydrogen as Water	238
2. Nitrogen and Oxygen as Nitric Acid	239
3. Sulphur and Sulphuric Acid	240
4. Phosphoric Acid	242
5. Chlorine }	244
6. Bromine	245
7. Iodine	247
8. Fluorine	248
9. Carbon and Carbonic Acid	250
10. Boracic Acid	251
11. Silicic Acid	252

EXAMPLES OF THE METHOD OF PROCEDURE employed in examining different Compounds for all their Constituents, with the aid of the Blowpipe :—

α. Salts :—

1. Sulphate of Potassa	254
2. Nitrate of Potassa	255
3. Iodide of Potassium	255
4. Carbonate of Soda	256
5. Chloride of Ammonium	256
6. Sulphate of Soda	256
7. Potassa-Alum	257
8. Bromide of Sodium	258
9. Sulphate of Magnesia	258
10. Chloride of Lithium	258
11. Celestine	258
12. Calcareous Spar	259
13. Pseudo Apatite	260
14. Chloride of Sodium	263
15. Borax	263
16. Phosphate of Lead	263
17. Nickel Ochre	265
18. Wolfram	266

β. Aluminates :—

Spinelle	267
-----------------	-----

γ. Silicates :—

1. Tabular Spar	269
2. Soap Stone	270
3. Pyrosmalite	270
4. Felspar	271

	Page
5. Bohalag	272
6. Black Copper Slag	274
3. Compounds of Metallic Oxides :—	
1. Brown Earthy Cobalt	275
2. Abstrich	276
e. Sulphides, Arsenides, and Selenides :—	
1. Zinc Blende	278
2. Cupriferos Sulphide of Bismuth	278
3. Bournonite	279
4. Nickel-Glance	280
5. Grey Copper	281
6. Rohstein	282
7. Lead Speiss	283
8. Cupriferos Selenide of Lead	286
§. Combinations of Metals which contain little or no Arsenic :—	
1. Native Amalgam	287
2. Workable Lead	287
3. Impure Tin	288
4. Black Copper	288
5. German Silver—Argentan	289

SECTION III.

DESCRIPTION OF A CONVENIENT AND USEFUL BLOWING APPARATUS FOR QUANTITATIVE ANALYSIS	293
---	-----

QUANTITATIVE ANALYSIS WITH THE BLOWPIPE.

Preparation of the Substance whose Metallic Constituents are to be ascertained	298
DESCRIPTION OF THE VARIOUS QUANTITATIVE ANALYSIS	299
I. THE SILVER ASSAY :—	

¶ A. Ores, Minerals, and Furnace Products :—

a. Examination for Silver in substances containing Volatile Ingredients	300
--	-----

CONTENTS.

XV

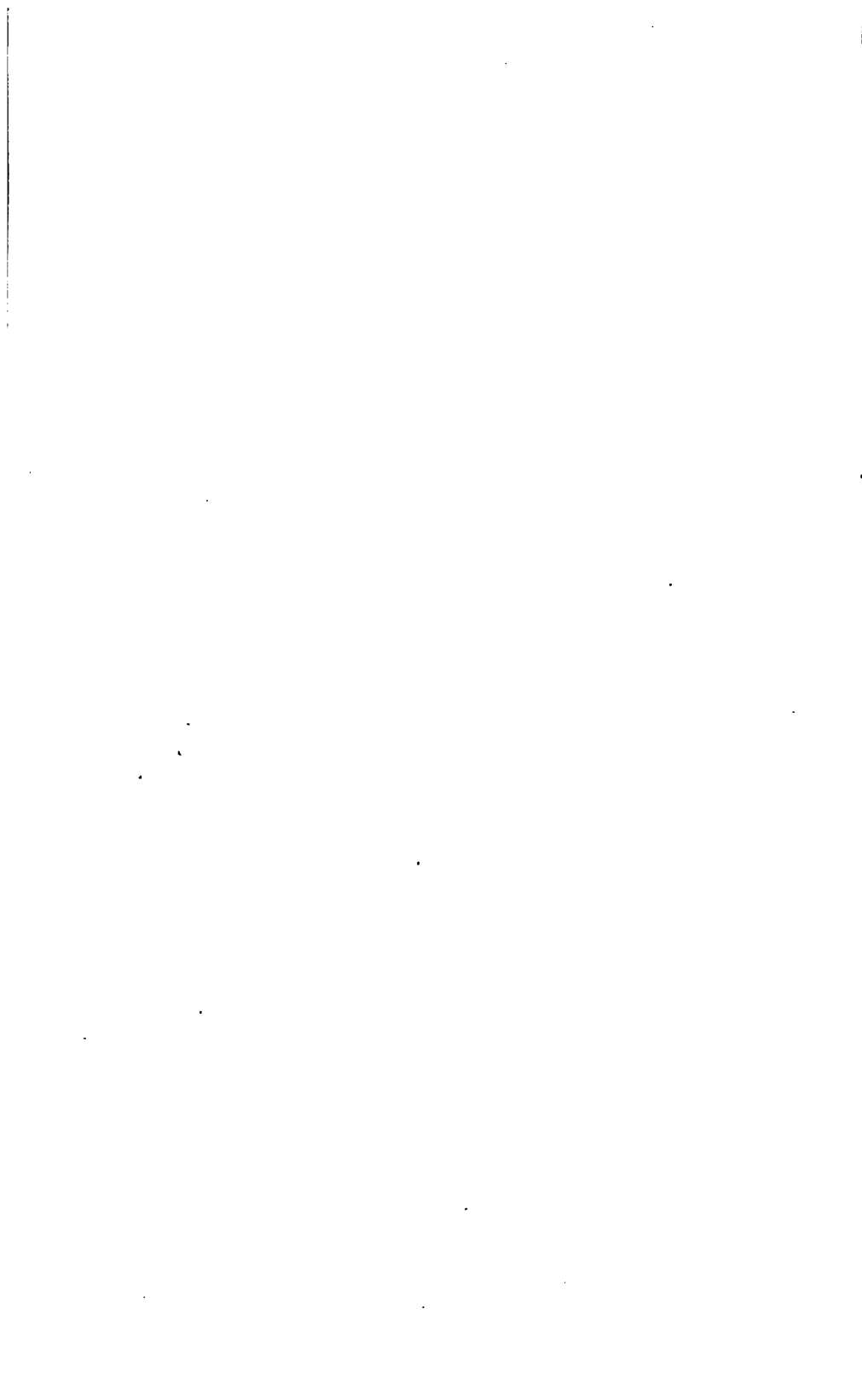
	Page
Weighing and Dressing of the Assay	301
Fusion of the Assay	303
Treatment of the Workable Lead obtained by the Fusion ...	307
The Oxidation	307
The Cupellation	309
Determination of the Weight of Silver Globules obtained ...	313
Compensation Table for the Loss of Silver sustained in Cupellation	315
b. Examination of Ores, Minerals, and Furnace Products containing no Volatile Ingredients except Chlorine	317
c. Examination of Furnace Products, consisting of Metallic Oxides which are easily reducible upon Charcoal	318
d. Examination of Minerals which are not decomposed by Borax or Lead upon Charcoal	319
¶ B. <i>Metallic Compounds</i> :—	
a. Examination of those Compounds which have Silver as a principal constituent	320
b. Examination for Silver in Metallic Compounds consisting chiefly of Gold	321
c. Examination for Silver in Alloys wherein Copper and Nickel predominate	321
d. Examination of Metallic Compounds, the predominating Metal being Lead or Bismuth	322
e. Examination of Metallic Compounds in which Antimony or Zinc prevails	324
f. Examination of Metallic Compounds in which Tin is the principal ingredient	324
g. Examination of Metallic Compounds the principal ingredient of which is Mercury	325
A. Examination of Metallic Compounds having Iron or Steel for a principal ingredient	326
II. THE GOLD ASSAY :—	
¶ A. <i>Ores and Furnace Products</i> :—	
Examination of Gold Ores, Auriferous Silver Ores, and Furnace Products containing Gold and Silver	328
¶ B. <i>Metallic Compounds</i> :—	
a. Examination of such as contain Gold and Silver only	332
b. Examination of Metallic Compounds containing other Metals besides Gold and Silver	334
c. Examination of Metallic Compounds consisting of Gold and Mercury	334
1. Determination of the Weight of Gold or Silver Globules by their Volumes	335
HARKORT'S Scale	335
PLATTNER'S Scale	338

	Page
2. Application of the Scale for determining the Weight of Gold Globules obtained in Quantitative Assayings	340
Table, adapted to HARKORT'S and PLATTNER'S Scales, of the content of Silver and Gold in an Assay-Globule, in loths, and centimals	342
 III. THE COPPER ASSAY :—	
¶ A. <i>Ores, Minerals, Products of Furnaces and Manufactures :—</i>	
a. Examination of such as contain Volatile Ingredients	344
b. Examination of substances containing the Copper in an oxidized state, either pure, or combined with Acids and Water, or vitrified with earthy constituents, <i>et cetera</i>	349
¶ B. <i>Metallic Compounds :—</i>	
a. Examination of Alloys of Copper and Lead	352
b. Examination of Alloys composed of Copper, Iron, Nickel, Cobalt, Zinc, and Bismuth, in which the Copper is combined with one or several of these metals, and frequently with lead, antimony, and arsenic	355
α. Affinage of the Impure Black Copper prepared on a large scale from Cupriferous Lead Ores	355
β. Affinage of the Black Copper obtained by the treatment, on a large scale, of Sulphide of Copper, Copper-slate, and non-plumbiferous ores in general	356
γ. Determination of the Percentage of Copper in German Silver, and in other Nickeliferous Compounds containing little or no Lead	357
c. Examination of Alloys of Copper and Antimony	359
d. Examination of Alloys of Copper and Tin	359
 IV. THE LEAD ASSAY :—	
a. Examination for Lead in Minerals, Ores, and Products of Smelting Works, containing this Metal combined with Sulphur :—	
First Method	362
Second Method	369
b. Estimation of the Lead in Minerals, Ores, and Artificial Products, containing this Metal in the state of Oxide, combined with Mineral Acids	372
c. Method of estimating the value of Lead in Products of Smelting Works, and other artificial substances, containing Lead in a pure oxidized state, or only combined with Vegetable Acids	374
d. Method of estimating the value of Lead, in Minerals containing this Metal alloyed with others	374
 V. DETERMINATION OF TIN :—	
a. Determination of Tin in Minerals, Ores, and Artificial Products, in which the Tin is combined with Sulphur	377
b. Determination of Tin in Minerals and Products, containing this Metal in an oxidized state	382
c. Determination of Tin in Stanniferous Alloys	383

APPENDIX.

	Page
SYSTEMATIC ARRANGEMENT OF THE OXIDIZED MINERALS, ACCORDING TO	
THEIR BEHAVIOR BEFORE THE BLOWPIPE	386
BEHAVIOR OF THE URINARY CALCULI BEFORE THE BLOWPIPE :—	
1. Uric Acid Calculi	391
2. Urates of Soda Calculi	392
3. Urates of Ammonia Calculi	392
4. Phosphate of Lime Calculi	393
5. Ammoniacal Phosphate of Magnesia Calculi	393
6. Calculi composed of the Phosphates of Lime and Magnesia— <i>Calculi fusibiles</i>	393
7. Calculi composed of Oxalate of Lime	394
8. Calculi containing Silica	394
9. Cystic Oxide Calculi	394
TABLE OF ATOMIC WEIGHTS	395
INDEX TO MINERALS	399
ERRATA	406

SECTION I.



SECTION I.

I.—THE BLOWPIPE.

THIS instrument was long employed in the arts, before any one conceived the idea of applying it to chemical experiments, performed in what is called the dry way. BERGMAN tells us, that the first person who so used it was ANDREW VON SWAB, a Swedish Metallurgist, and Counsellor of the College of Mines, about the year 1733. He left no work on the subject, and it is unknown to what extent he carried the researches which he made with this instrument. CRONSTEDT, who laid the foundations of Mineralogy, and whose genius so outstripped the age in which he lived, used the Blowpipe to distinguish mineral substances from one another, by means of fusible reagents. From their action such modifications on the objects to which they were applied resulted, as led to some conclusions respecting their composition, and served as a basis for the classification he adopted. In his time the intercourse between men of science was by no means so open as at present; the discoveries of one man were seldom communicated to his fellow-laborers, and each pursued his researches with no other help than the experience of the generation which had passed away, and become in some measure public property. At such a period, CRONSTEDT carried the use of the Blowpipe to a degree of perfection, that can only result from persevering industry; but, as slight services in the cause of science were not as yet honored with general attention, he who at first was afraid to make himself known as the author of that system of Mineralogy which has perpetuated his memory, still less thought of describing in detail this new application of the Blowpipe, and the system he adopted. He only published such results of his experiments, as might serve to distinguish minerals from each other, by affording characters

peculiar to the different species. VON ENGESTRÖM, who published an English translation of CRONSTEDT's system in 1765, annexed to it a treatise on the Blowpipe, in which he particularly noticed the processes of the author, as well as the principal results of their application to the minerals then known. This treatise did not appear till 1770, and was translated and published in Swedish by RETZIUS, in 1773. It attracted the general attention of Chemists and Mineralogists to the use of this instrument; they, however, derived at first little other advantage from it, than as a means of ascertaining the fusibility of bodies, and occasionally their solubility in borax; for it was difficult to form a just estimate of its value, in consequence of the want of that skill in its application, which can alone be derived from patience and practice, together with a sufficient knowledge of the phenomena presented by the various substances used as fluxes; whilst the difficulties attending its use were sufficiently evident; and hence, everywhere but in Sweden, the art of the Blowpipe made little progress. As in other practical sciences, books alone are weak masters to make proficient; but they who had seen the manipulation of CRONSTEDT and VON ENGESTRÖM, learned to work like them, and transmitted their skill to their successors. BERGMAN went further than CRONSTEDT; he extended the use of the Blowpipe beyond the bounds of Mineralogy to the field of inorganic Chemistry; and in his hands it became an invaluable instrument for discovering very minute portions of metallic matter in analytical researches. His work *De Tubo Ferruminatorio* was first published at Vienna, in 1779. BERGMAN, on account of his health, was assisted in his experiments by GAHN, who particularly applied himself to the use of the Blowpipe in his mineralogical studies, in consequence of the readiness with which it affords decisive results. The operations which he performed under BERGMAN's inspection, who caused him to examine all the minerals then known, taught him how each conducts itself before the Blowpipe. Assisted by the experience which he thus acquired, he continued to employ the instrument in every kind of chemical and mineralogical inquiry; whence he attained such a degree of skill in its use, that by its means he could detect in any body the

presence of substances, which had escaped the most careful analysis conducted in the moist way. Thus when EKEBERG asked his opinion respecting the oxide of *Tantalum*, then recently discovered, and of which he sent him a small specimen, GAHN immediately found that it contained tin, although that metal does not exceed one per cent. Long before the question was started, whether the ashes of vegetables contained copper, BERZELIUS saw him many times extract, with the Blowpipe, from a quarter of a sheet of burnt paper, distinct particles of metallic copper. GAHN always travelled with his Blowpipe, and the continual use which he made of it led him to several improvements in its application ; he examined a great number of reagents, in order to find new methods of arriving at the knowledge of certain substances, and the whole was imagined and executed with such sagacity and precision, that his results were entitled to the greatest confidence. He most readily and carefully instructed those who were desirous of information on the subject ; but he never appears to have thought of publishing an account of his labors, nor has it been done by others. In all the rest of Europe, only one distinguished Naturalist applied himself to the study of the Blowpipe and its uses, and submitted a large number of mineral substances to its action. This was SAUSSURE, justly celebrated for his geognostic researches on the Alps of Switzerland. He, as well as CRONSTEDT, employed the Blowpipe chiefly in distinguishing minerals ; and although he made additions and improvements on the subject, he ranks far behind GAHN in respect to the results which he obtained.

The science of the Blowpipe, like all others, still continues to unfold new secrets, and offers more beautiful advantages in the way of analysis ; for the use of this instrument is not confined to the detection of metals in minerals only, but by its aid the percentage of many of these, especially the precious ones, may be ascertained with surprising accuracy. HARKORT was the first to draw attention to this course of metallurgic determinations ; he published the result of his labors in 1827, but it is to the diligence and ingenuity of the German author of this dissertation that the quantitative determinations by the Blowpipe owe their great exactness and simplicity.

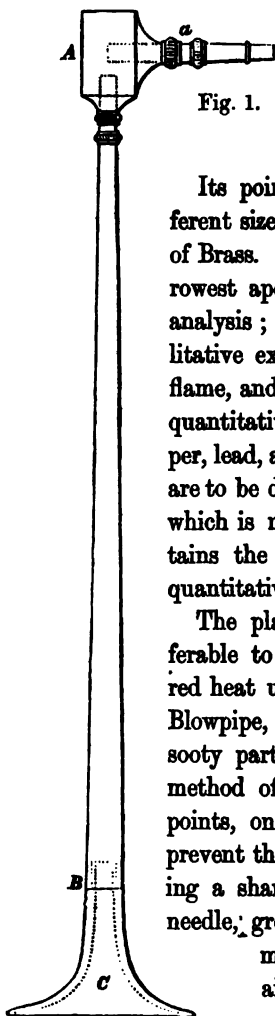


Fig. 1.

The Blowpipe is generally made of brass. Fig. 1 represents the one described and used by GAHN, and approved of by BERZELIUS.

Its points are turned and bored of three different sizes, and are partly of Platinum and partly of Brass. The first of platinum, contains the narrowest apertures, and is employed for qualitative analysis ; the second, of brass, is used for such qualitative experiments as require a strong oxidation flame, and for heating silver, gold, and copper, in quantitative assayings, also for the roasting of copper, lead, and tin ores, the metallic contents of which are to be determined with precision ; and the third, which is manufactured of the same material, contains the widest bore, and is for obtaining the quantitative proportions of lead and tin.

The platinum points, however, are always preferable to those of brass, because by a moderate red heat used on charcoal before the flame of the Blowpipe, they are more easily cleaned from the sooty particles obstructing their apertures. This method of cleansing cannot be applied to brass points, on account of their rapid oxidation ; to prevent this, the operator must adapt to the opening a sharp-pointed fragment of horn, or a small needle, ground to one-half of its length ; by this

means, the aperture through which the air passes is purified. The black matter, adhering to the exterior of the tube, can

be easily removed with a small wooden or leaden file, upon which is placed a quantity of fine moistened bone ash.

To defend the lips from injury, a mouthpiece of horn—c—is affixed to the Blowpipe. If, during the act of blowing strongly, this mouthpiece be pressed to the lips, partially open a much greater blast can be obtained than otherwise ; and when

an operator once becomes accustomed to its management, his lips will not be fatigued. All who employ this instrument should have a suitable mouthpiece.

A Blowpipe entirely of silver is not to be recommended for quantitative researches, because at such experiments the manipulator must often blow uninterruptedly for several minutes, and the silver, being a good conductor of heat, becomes so hot that it cannot be held between the fingers, unless enclosed in a nonconducting substance.

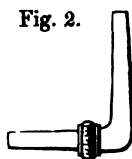


Fig. 2.

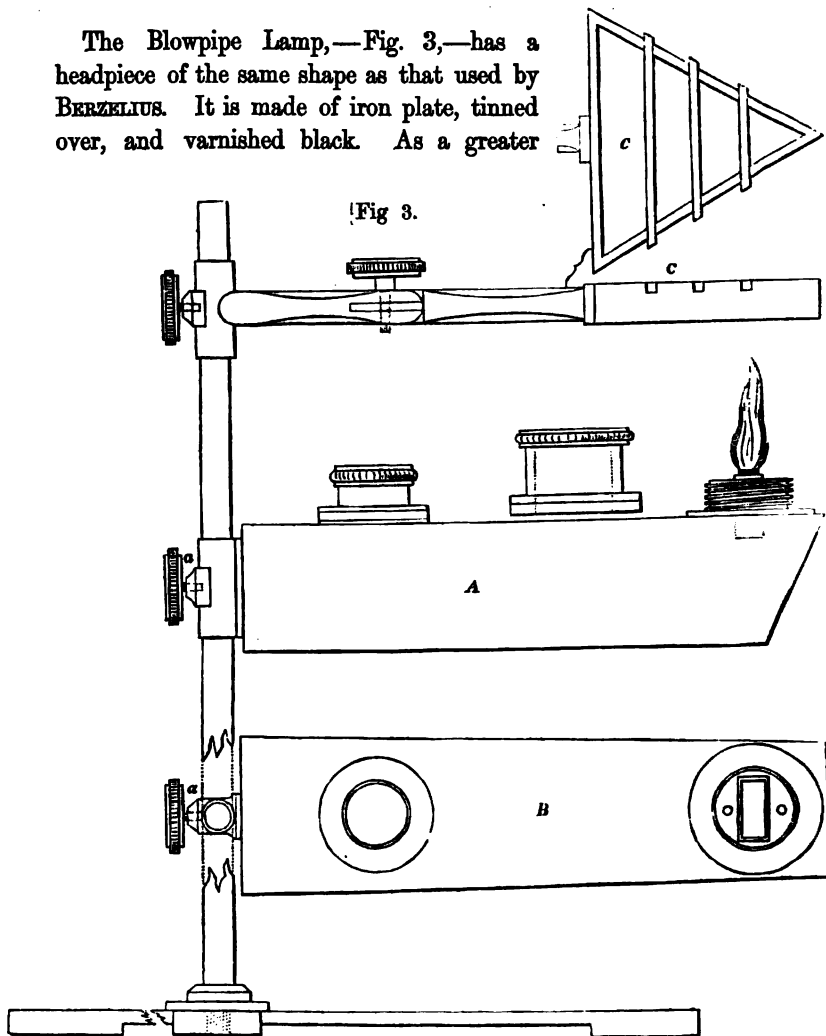
When the Blowpipe was required for glass, *BERZELIUS* employed a tube bent to a right angle ;—see Fig. 2. This tube will be found convenient, as it can be turned in many directions, to suit the materials operated upon.

When the hands are required for examinations, the Blowpipe should have affixed a very broad mouthpiece of bone ; as by this means it can be held safely.

II.—THE COMBUSTIBLE MATERIAL.

In many qualitative examinations with the Blowpipe, an operator may employ the flame from wax, tallow, or an oil-lamp. These, however, offer very great inconveniences in consequence of the carbonaceous matters which they hold mechanically being deposited upon the substance under examination, thus depriving the operator of the opportunity of making any notes as to change of color or the like ; besides, the heat they afford is never so high as to answer the requirements of the assayer ; even the gas flame, which is the most convenient for Blowpipe operations, cannot be used naked, but after a sufficient volume of air is mixed with the gas, to oxidize its carbon, this mixture, on being ignited, answers every purpose, and is now generally resorted to in all laboratories.

The Blowpipe Lamp,—Fig. 3,—has a headpiece of the same shape as that used by BERZELIUS. It is made of iron plate, tinned over, and varnished black. As a greater



proportion of substance is used in a quantitative than in a qualitative analysis, and also a stronger heat is required, the socket in the lamp is wider than usual, so that the flame of the Blowpipe may be deflected to any desired point,—Fig. 6, p. 11.

The brass ring encircling the socket, must not be too distant or elevated, as is the case with those lamps which have the top screwed to the ring. In this lamp, the screw is affixed to the external part, so that a cover can be placed over it, which, below, is provided with a large margin of brass, having fastened to it a piece of leather, which can be placed over it. The leather is attached in the following manner:—First, that side which is *not* to be cemented to the brass rim, must be covered over with molten wax, and when it hardens, the other side must be varnished with a strong solution of shell-lac in alcohol; over the brass edge rub also some of the solution, and then keep the leather pressed upon it for some time. When the spirit has evaporated, the shell-lac remains as a durable cement. The screw in the lamp does not permit a drop of oil to escape.

If a separate opening for pouring in the oil be made at the upper part of the lamp—*b*—according to the proposal of HARKORT, it must be wide enough to enable the operator to ascertain how high the liquid reaches. A screw can also be adapted to this opening, as the figure in the drawing shows.

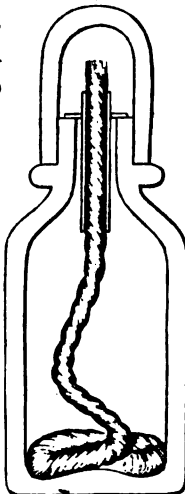
The wick must be cotton; and, before it is immersed in the oil, it should be folded together three or four times, and then cut parallel with the oblique side of the socket to allow the flame to be easily directed downwards.

The lamp for experiments may be placed on the brass stand *A*, and fastened by a screw *a*, to the brass pillar.

On the same apparatus may be adapted a brass triangle *C*, containing three bars of different lengths, and a movable arm. This triangle serves as a support for drying or fusing any article over the flame of an oil or spirit-lamp.

A small spirit-lamp may be profitably used for heating substances, containing volatile ingredients, in glass tubes or alembics, and for melting various assays with bisulphate of potassa in the platinum spoon, *et cetera*. For

Fig. 4.



this purpose *BERZELIUS* used a small phial of strong glass, with a ground top, in which is inserted a tube of silver or iron, for containing the wick. Fig. 4 shows a vertical section of such a lamp, which is about one and a half inch in diameter. Although a small brass lamp of another form is sometimes used, still, the glass one, if carefully handled, is in every respect preferable.

III.—THE BLOWING, AND DESCRIPTION OF THE FLAME.

The air of the Blowpipe is obtained from the mouth, and not from the respiratory organs, because they could not, without fatigue, afford a sufficient stream. The current of air is forced through the tube by the action of the *Musculi Buccinatores*, which are in the cheeks. During the blast, the communication between the chest and mouth is closed, so that respiration is carried on through the nostrils.

Until a person is accustomed to the use of this instrument, a strong and uninterrupted stream cannot be obtained; but after a few days' practice the process becomes easy. The fear of blowing being injurious to health is soon removed.

Fig. 5.



To obtain an intense heat by blowing on the lighted combustible, requires a previous knowledge of its various parts; for on examining the flame of a lamp which does not emit smoke, it will be found divided into many. This appearance is more clearly to be seen in the flame of a wax or tallow candle, where four distinct portions are noticed. Fig. 5 represents a flame: a fine clear blue color is visible at *a b*; this azure tinge surrounds the burning wick, but becomes less distinct as it ascends, and, where the flame elongates perpendicularly is wholly diffused. In the centre of the flame is a very dark conical portion *c*; this part is encircled by *d*, the sphere whence nearly all the light emanates: external to this is *a, e, b*, which part affords scarcely any light, but gives out more heat than any other. The hottest part of this exterior flame is at *f*; the heat diminishing towards the

summit *e*, and base *a b*. When a very fine iron wire is introduced horizontally into the flame, it expands and becomes of a white heat, at the outer part *a, e, b*, whilst at *c* it barely assumes a red one.

The cause of this phenomenon is as follows:—The small interstices of the wick, like other porous bodies, imbibe the melted tallow or wax, which is a combination of carbon, hydrogen, and oxygen; these by heat are converted into an empyreumatic oil—two of the compounds of carbon and hydrogen, carbide of hydrogen and olefiant gas, and carbonic oxide, which burn and produce flame. The external part *a, e, b* is the hottest, because it is in contact with the atmospheric air which supplies oxygen. The small blue margin is produced from combustion of carbonic oxide and a trifling proportion of carbide of hydrogen. The inner dark cone is filled with combustibles which are only partially consumed, on account of the air which passes through the flame being deprived of its oxygen by *a, e, b*.

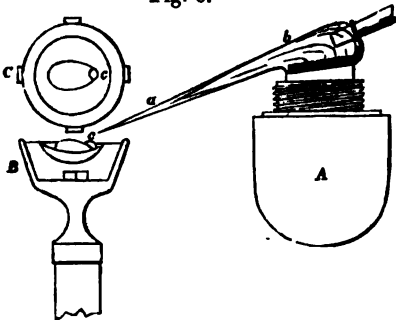
Of the four different parts of the flame, three are as easily perceived in the flame of a lamp as in the flame of a candle; but the last *a, e, b*, if at all seen in the flame of the latter, must be very indistinct, as it only disperses a small quantity of light.

The following are the three parts necessary for researches with the Blowpipe:—the blue, *a, b*; the one which produces much light, *d*; and the outer, *a, e, b*. As the first and second are the most essential, they will be described at length.

THE OXIDATION FLAME.

When an operator with the Blowpipe blows lengthways on the

Fig. 6.



flame of a lamp, so that the air just passes over the wick, Fig. 6, a long narrow blue flame will be formed,—which is the same as *a b*, Fig. 5, excepting in shape;—the flame of Fig. 6 is conical, that of Fig. 5 oval. The heat is greatest at the

extremity of this flame, because it is there the largest quantity of air sent through the Blowpipe is consumed.

In the free flame, the warmest part forms a girdle round the whole, the heat being most intense at *f*. Where the heat is greatest, substances which are not acted upon by an ordinary flame, oxidate, melt, and evaporate. The further the matter can be kept from the apex of the blue flame, in a temperature sufficiently high, is favorable to oxidation, because the atmospheric air will have free access. The operator must not blow too strongly, as it would be very deleterious to oxidation, especially if the assay rests on charcoal. The wick must be evenly cut, and kept free from all fibres, otherwise yellow streaks, which have a reducing effect, will be mixed with the blue flame. If these directions be adhered to no difficulty can be found in ascertaining how far the tube should be introduced to produce the oxidating flame. It will be found most serviceable, not to have the opening too small in the point of the Blowpipe.

THE REDUCTION FLAME.

By allowing a stream of air to pass over the wick, at a greater distance than in the production of the oxidating flame, the whole ignited mass receives the same direction, and appears as a long narrow luminous cylinder, the end being surrounded by that part of the flame which emits only a small degree of light. By forcing the air as described, the particles rising from the wick, which are perceptible in a free flame, are consumed, and the heat becomes concentrated in a less space. If such a flame be directed upon a metallic oxide, so that the point covers it totally, the oxygen will be eliminated, either partially or entirely, according to the stability of the compound, or the materials upon which it is exposed,—charcoal, or platinum wire.

If the reduction of a peroxide into a protoxide be attempted on a platinum wire, it will succeed, completely, only when the assay is surrounded with the luminous part of the flame: this effect can also be produced in the blue part of the flame, with easily reducible metallic oxides, if the accession of atmospheric air be prevented,

and the substance rest on charcoal. When the oxide of lead is mixed with a borax-glass, it can be reduced to the metallic state, upon charcoal, with as much facility in the blue as in the bright part of the flame. Even if a piece of tin be liquefied upon charcoal in the blue part of the flame, no oxide is formed, because oxidation cannot take place when atmospheric air is excluded from the surface of a molten mass. The foregoing results avail for some, but not for all reducing tests upon charcoal. Positive indications are attainable only in the bright part of the flame. The reducing flame is much more difficult to form than the oxidating; for this reason, the following directions must be studied.

a. The wick should not be drawn too high out of the holder, nor must it be too short; because in the first case a smoky flame would ensue, impeding reduction, from the deposition of soot upon the assay, and in the second, the substance could not receive sufficient heat, if surrounded by the luminous part of the flame.

b. To ensure exact results, have the wick evenly cut, and perfectly free from all extraneous fibres, as in the production of the oxidating flame.

c. To secure success, when the reducing flame is produced, let it be kept uninterrupted.

And lastly,—

d. The operator must use great precision as to the treating of a qualitative or a quantitative analysis. For qualitative examinations, a Blowpipe with an aperture narrower than that for oxidation is employed; but for quantitative researches, which should be always performed on charcoal, a Blowpipe with an opening rather wider is necessary. As, in many qualitative analyses, the flame has often to be altered, and a different point of the Blowpipe affixed, it is better to employ a point with a narrower orifice, because, with one of too large a calibre, oxidation is more easily effected than reduction.

IV.—THE SUPPORTS.

1. CHARCOAL.—When a substance requires melting, it should be exposed to the flame of the Blowpipe upon a body that will

augment the heat, without combining with the assay. The best substance to employ for this purpose is well-burned pine. It should be cut with a saw into long pieces, partly in the shape of a parallelopiped, and partly four-sided like a prism. For qualitative analysis use the long sides of the charcoal, where the annular rings are on the margin ; and for quantitative use the interior part.

2. **PLATINUM WIRE.**—A moderately strong wire of platinum, about two inches long, and curved at one end, may be employed

Fig. 7. in many qualitative examinations with great advantage.—Fig 7, A.—The curve serves as a support in all tests of oxidation and reduction, where alteration of color only is to be observed. This support can be relied on, for it is totally free from the false variations of color which are too often perceptible when the assay rests on charcoal. In the treatment of compound metals, or in such tests of reduction where a body easily melted is to be operated upon, charcoal must, however, be used. It is necessary to have at hand several platinum wires, so as to proceed without being obliged to take off with force the glass adhering, after the extraction of the bead, or to wait for its solution in hydrochloric acid,—which is the better mode.

Should it happen that the platinum curve melts with the reagents, it must be cut away, and a new one formed. A wire can be used for a very long time, and when it becomes too short to be held between the fingers, the straight end may be fastened into a cork, or a hilt containing a box for holding the rest of the platinum wires, until any of them are required.—See Fig. 7, B.

3. **PLATINUM FOIL.**—The use of platinum foil in qualitative examinations is very limited, but in some cases it may be successfully employed. Foil of thin drawn platinum, a few inches long, and half an inch broad, should be selected. This metal is such a bad conductor of heat, that when the assay is exposed to the flame of the Blowpipe at one end, the other may still, without inconvenience, be retained between the fingers. Bodies in



the metallic condition which are easily fused or reduced, should seldom be tested upon platinum foil, because, from its combining with them, a hole might be produced in the metal. Should this occur, the hole can be lopped off with a scissors, and then, if the platinum be found too short, it can be easily held by forceps. Salts, or substances containing manganese, can in general be melted upon platinum foil; by this means the alkaline and earthy salts are distinguished, and the presence of manganese is recognised by the dull green color communicated to soda, upon cooling.

4. GLASS TUBES.—When the volatile ingredients contained in ores and minerals were to be ascertained, BERZELIUS employed a glass tube, from three to six inches in length, a quarter of an inch in diameter, and open at the ends. The body to be analyzed is inserted at one end, and heat applied to that part of the tube. If the compound requires only a slight degree of heat, the flame of a spirit-lamp will suffice; on the contrary, if a strong heat is necessary, the flame of the Blowpipe must be used. The volatile bodies, liberated during the roasting, sublime in the upper part of the tube, where they can be examined.

5. GLASS FLASKS.—If the quantity of water, or gaseous ingredient, contained in any substance, be required, or if a body, when heated strongly, decrepitates, a glass flask must be used, of the form of Fig. 8. If the small alembic be sufficiently wide for the free circulation of air, the vaporized bodies will be easily obtained. Flasks must not be employed in the sublimation of sulphur, arsenic, *et cetera*, lest combustion should ensue.

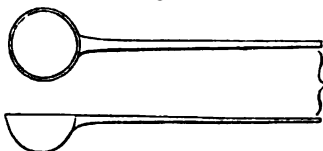


A supply of glass tubes and small flasks should always be in reserve. After the roasting has been performed in a tube, the part containing the fused substance should be removed with a file, and the remainder of the tube cleansed with the aid of a spirit-lamp and a wire enveloped in soft paper. When the tube becomes too short, one end can be sealed, and it may be used for tests of sublimation only.

6. TWO PLATINUM SPOONS.—The form of these utensils is

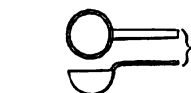
shown in the annexed woodcuts. The diameter of Fig. 9 is

Fig. 9.



9-16ths, and that of Fig. 10 3-8ths of an inch. The larger spoon has a handle of platinum, joined by rivets of the same metal, which can be encased in wood; the small one, when heated, may

Fig. 10.

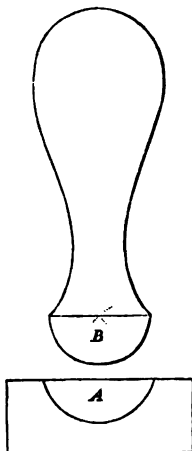


be held with pincers.

The first is used for melting various substances with bisulphate of potassa, and for heating gold obtained in quantitative assays; and the second, for fusing substances with nitrate of potassa. In some cases, when bodies containing water are heated with nitrate of potassa, the platinum spoon becomes tarnished with the remaining particles of metallic oxides, these may be removed, either by a drop of sulphuric acid, or by a hot solution of bisulphate of potassa. Rubbing the platinum spoon with charcoal powder is however the most effectual method of removing stains.

7. CLAY BASINS.—In quantitative determinations, basins are employed for roasting the Ores, and for the reduction of the lead

Fig. 11.



and tin oxides contained in calcined and uncalcined minerals, *et cetera*. These dishes are formed in the following manner:—at first a fire-proof clay is kneaded into a stiff paste with water, and then the forming surfaces of the press, which are of box-wood, —Fig. 11, A B,—are rubbed over with a paint-brush dipped in oil. The width of A at the upper part, is 7-8ths of an inch, and the depth 5-16ths; and B is constructed of a diameter smaller by the 32nd of an inch. Over the middle of the concavity of the press, place a slip of paper, three inches in length, and a quarter of an inch in breadth, and upon this a small clayball about half an inch in diameter.

Then take part B of the press, and stamp it horizontally upon the

clay mass lying in part A, as far as is required. This being accomplished, the superfluous clay will have exuded, and B can be removed easily, by careful turning. With a small knife, cut away the clay which is driven out, and then examine the margin, to learn if the basin be uniform throughout, and sufficiently thin.

Fig. 12 shows the cross section of one of these basins, which is only one thirty-second of an inch thick. Fig. 12.



If the basin be thicker in one place than in another, B must be applied with greater force to that part. When the basin is ready to be taken from A, the convex part of the press must be carefully removed, and then the slip of paper gently pulled, to extract the clay dish. The utensil, with the slip of paper adhering, is now put away on a stone to dry, and the press is ready for the formation of others.

An operator unused to this work will find that sometimes the basins alter in shape after leaving the mould; but this can be remedied by pressing them with the fingers upon the convex part, B.

A few hours after drying, the slip of paper falls from the clay dish. When this occurs, the basin must be heated to redness over a fire, in a potter's vessel, or in a platinum crucible over a large spirit-lamp.

In calcination, when these vessels are used, one may often serve as a lid to the other; but, for this purpose, their edges must be ground with a file.

No labor should be spared in manufacturing these basins so as to make them sufficiently thin. The clay must be made neither too stiff nor too moist; the first fault will prevent the vessel assuming a proper form, and the second endangers its falling to pieces when removed from the machine. The true consistence of the clay can be ascertained by two or three experiments.

8. CLAY CRUCIBLES. — These crucibles are employed with advantage in the performance of quantitative lead assays, and in cases where the compound to be examined consists of sulphides, arsenides, and selenides. They are easily manufactured with an iron instrument, consisting of a mould and a stamp; the mould being composed of two parts, united by a ring.

Fig. 13.

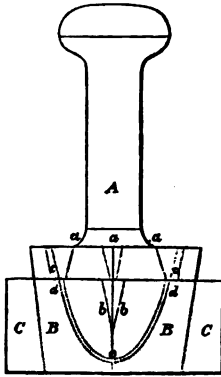


Fig. 13 gives the form of this instrument. The Stamp, A, is so bored through at four parts, *a*, that the internal diameter of each orifice is little more than the diameter of the side: but the cross section of the outer part is nearly the double. These perforations serve for extracting the superfluous clay that has been placed in the mould for the formation of a crucible. The forming part is three-quarters of an inch in diameter at the top, and five-eighths of an inch long.

The Mould, B B, consists of two parts, accurately joined, which form at the outside a truncated cone. At the inner side, the distance of each part is one thirty-second of an inch from the Stamp; and the edges near *b* are flattened, so that small depressions may be produced in the two opposite points of the mould, to prevent the crucible from being injured when the stamp is withdrawn. *c c* is the binding ring into which the mould is fitted, so that, when unclapsed, the mould falls with it exactly into a plane.

For the formation of these crucibles, a stiff paste is prepared with fire-proof clay; when this paste acquires a certain dryness, it is worked with the fingers into small balls, each containing more clay than is required for the manufacture of one of these vessels. Before introducing one of these balls into the mould, the forming parts and the stamp are well smeared with oil, by means of a fine brush, and then the mould, with the binding ring, is placed on an anvil resting upon a folded cloth to give elasticity. When the machine is thus arranged, the stamp is struck perpendicularly upon the ball with a mallet, until the protruding part, *c c*, rests on the margin, *d*, of the mould. The stamp causes the clay in the mould to spread, and the superfluous portion escapes through the small apertures at *a*. After the stamp has remained in the mould for about five minutes, it is gently screwed out; by this means the clay contained in the openings, *a*, and which is still in conjunction with the crucible, is smoothly removed.

To extract the crucible requires great care. The binding ring is first taken off the mould, and then the two sides *B B* are slightly struck with a hammer ; this causes them to separate and fall open at *b b*, and the crucible remains perfectly detached, upon *a*. The crucible, when cleansed from adhering clay by a small knife, is dried and heated in the same manner as the clay basins.

9. SODA PAPER.—In heating the quantitative silver assays before the Blowpipe, HARKORT found it necessary, instead of laying the ore, mixed with reagents, upon charcoal, to enclose the mixture in a substance that will resist the first effects of the flame of the Blowpipe, and prevent any loss of the particles. After some investigations, he ascertained that fine letter-paper, saturated with a solution of soda, was the best. This paper, which is made use of advantageously, not only in silver, but also in many more quantitative analyses, is prepared in the following manner :—In a flat basin, dissolve half an ounce of crystallized carbonate of soda, perfectly free from any sulphate, in one ounce of distilled water ; then steep a number of strips of fine letter-paper in the solution for a few minutes, and allow them to dry in the open air. When dry, cut them into pieces, one inch and three-eighths long, and seven-eighths of an inch broad.

10. A MIXTURE of *seven parts of Charcoal with one part of Clay*.—This mixture is used for lining the clay basins in which Lead and Tin ores are to be calcined. It is best prepared as follows :—Weigh seven parts of fine dry charcoal powder, and one part of finely sifted clay ; mix the latter with water, and then add the former ; next, knead the mixture into a paste. The tenacious mass thus formed is dried in a warm stove, and reserved for use. When a basin is to be lined with this mixture, a small quantity of it must again be moistened, and strongly rubbed over the interior of the vessel employed for calcination. The thickness of the lining at the bottom of the basin must be about one sixteenth of an inch, and at the top rather less. When this operation is finished, the clay dish is placed upon the triangle, *c*, Fig. 3, and dried over a spirit-lamp. Fig. 14 shows the cross section of such a lined basin.



Fig. 14.

V.—INSTRUMENTS, ET CETERA, REQUISITE IN ANALYSIS WITH THE BLOWPIPE.

1. A BALANCE.—The operator can please himself as to the form and quality of the balance. It may, however, be useful to give a few rules which will be applicable in the choice of it, and then point out the methods for testing its accuracy.

It should be of sufficient strength to bear from seventy to eighty grammes on each scale, and must be enclosed in a glass case to protect it from dust. This case must not be too small, and especially its sides should not approach too near the beam; the anterior wall should be divided into three parts, the centre one being fixed, and the two ends opening as sashes.

The balance must be provided with a proper contrivance to render it immovable whilst the weights are being placed on the pans. The best arrangement is that which raises the fulcrum and end edges from their supports, keeping the scales steady at the same time. It is most convenient when the operator can perform this part of the process without opening the case. It is necessary that the balance should be provided with an index to mark its vibration, and it should have a pendulum, or two hydrostatic scales for placing the edges exactly on a horizontal level; for this purpose it is best that the case should rest upon screws.

For weighing the milligramme and its fractions, the beam should be divided into ten equal parts accurately marked; on these marks or points a centigramme crotchet or hook is suspended instead of placing the weight on the pan.

The beam should have a screw to adjust the centre of gravity, and also two screws to regulate the length of the arms.

The following experiments serve to test the accuracy and sensibility of a balance.

a. Should the scales not be perfectly equal, they are accurately adjusted by means of tinfoil, and a milligramme weight is placed in one of them. A good and useful balance must distinctly turn

with this weight ; a delicate chemical balance should indicate even 0.1 milligramme.

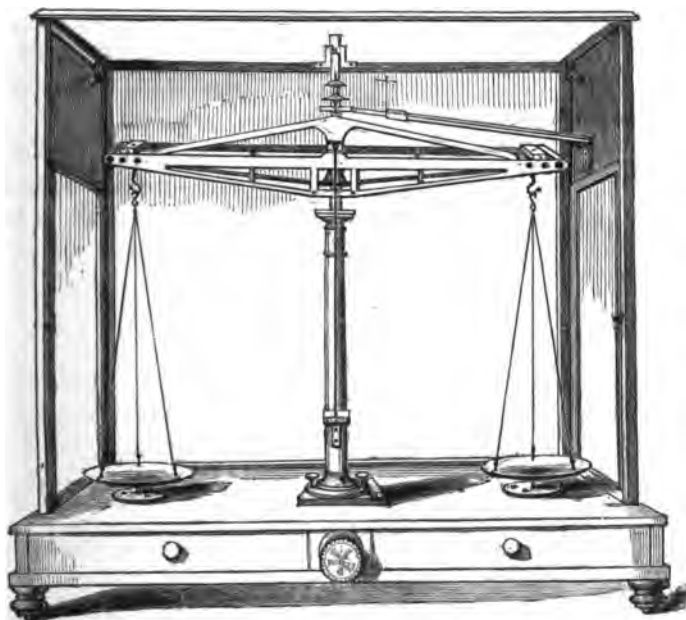
b. When both pans are loaded with the maximum weight the construction of the balance will admit of, and a milligramme is added to the weight in one scale, the beam ought to turn to the same degree as when the pans are unloaded.

c. Both scales are equally loaded with, say fifty grammes ; the weights are then interchanged so that those of the right hand pan are transferred to the left, and *vice versa*. A balance, the arms of which are perfectly equal, must maintain its equilibrium upon this exchange.

The balance is set in motion, then arrested, and subsequently set in motion, until it recovers its equilibrium. If its quality is good, it will invariably assume the original position.

Fig. 15 represents an OERTLING'S balance, which is the one generally used.

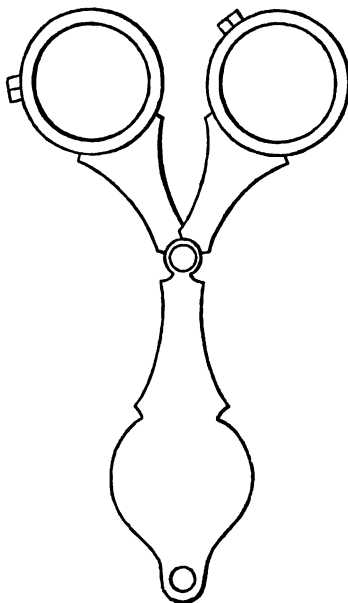
Fig. 15.



2. A SET OF WEIGHTS.—Weights, above 100 milligrammes, are seldom required. It is best, however, to have a box containing the Gramme and its divisions.

These, to prevent their oxidation and the adherence of foreign matter, should be kept in close boxes, well lined. They should on no account be touched by the fingers, but lifted, when wanted for use, with a pair of forceps.

Fig. 16.



3. A MICROSCOPE is indispensably necessary, in various operations to distinguish the effects of the flame and the diameter of metallic globules. An instrument formed of two fine plano-convex lenses—Fig. 16—of equal magnifying power, is very suitable; they must be so arranged that, when required, one can slide over the other, by a movable arm.

4. FORCEPS.—Various kinds of Forceps are needful for experiments with the Blowpipe :—

a. One with platinum points, to hold a small lamina of a specimen in the flame in order to try its fusibility—Fig. 17.

Fig. 17.



b. One much broader and stronger than a pincers, for detaching small fragments from a specimen, without the formation of much dross. Fig. 18.

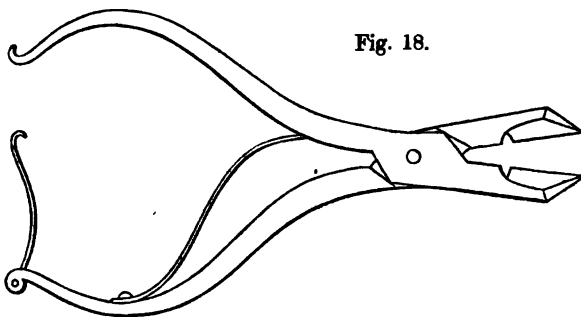


Fig. 18.

c. One of steel—Fig. 19—is employed for removing the scoræ in silver and gold assay-

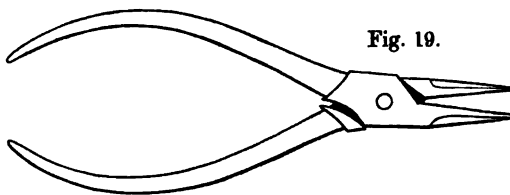


Fig. 19.

ings, and in various other operations. Its points must be broad, and its inner surfaces rough.

d. One of brass, for holding small fragments of minerals, *et cetera*, in the flame. Fig. 20.

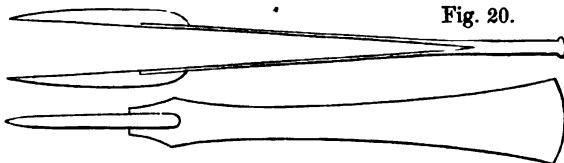


Fig. 20.

e. One of brass, with points exceedingly fine, for raising the weights, *et cetera*, and placing the silver and gold globules upon the graduated scale ; and,

f. One of iron, for trimming the lamp wicks.

5. A MORTAR AND PESTLE of *Agate* ; or of *Calcedony*, which is preferable.—This should be about $2\frac{1}{4}$ inches in diameter, $\frac{3}{4}$ of an inch high, and $\frac{7}{16}$ ths of an inch deep, transparent at the bottom, and free from fissures : they are not only applicable to the pulve-

rization of substances that are to be qualitatively examined, but also for purifying the scorise and coaly particles of reduced metallic globules. When very hard substances are triturated in a mortar similar to this, small cracks may appear, which the particles of the minerals fill, and produce false results. To guard against these, an operator, before each examination, should levigate some fine moistened bone-ashes in the mortar, which will be found completely to fill all the small crevices. GAHN having once lost the pestle of a mortar similar to the one above described, took a button of calcedony, and fastened it with sealing wax to a cork. This new pestle answered so remarkably well, that it was the only kind he ever used afterwards. BERZELIUS states that he was obliged to have recourse to a similar expedient.

6. A HAMMER.—This instrument should be of hardened steel, similar in shape to Fig. 21. When the broad sharp end becomes blunt from continued usage, it may be easily repaired by applying it with force to the grinding-stone.

7. AN ANVIL.—A polished anvil, of hardened steel—Fig. 22—will be found exceedingly useful in the laboratory, for crushing minerals, ores, *et cetera*. It should be in the form of a paral-

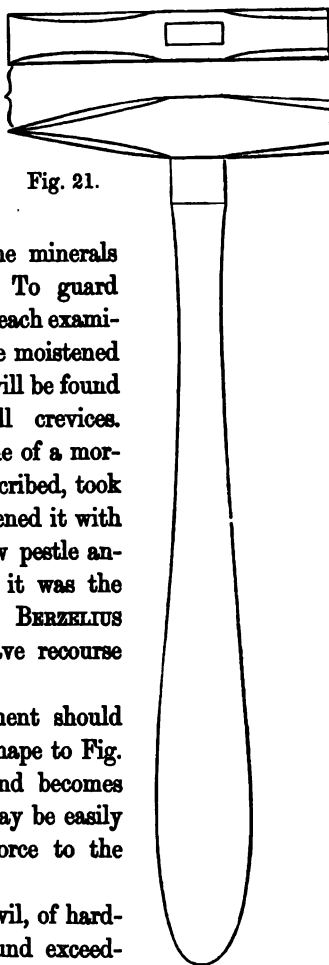


Fig. 21.

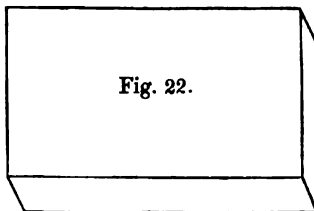


Fig. 22.

lelopiped; about $2\frac{1}{4}$ inches long, $1\frac{3}{8}$ inch in diameter, and $\frac{5}{8}$ of an inch in thickness. When a substance is to be broken into fragments, it must first be wrapped in strong paper, placed upon the instru-

ment, and then struck sharply with the hammer. Should the paper, in this process, be torn, and thrown off the face of the anvil, the malleable particles will be found adhering to it, and may be examined separately.

If a mineral is to be heated upon charcoal before the Blowpipe, it can be reduced to small pieces in a machine similar to that below. As it is frequently employed in the quantitative examination of minerals, it will be described :—Upon a hardened steel plate, *AB*,—Fig. 23,—is a depression *C*, containing a hollow cylinder, *DE*, which is completely filled by a massive cylinder *F*, hemispherical at the end. The two cylinders, *DE* and *F*, are also hardened steel, well ground. When a mineral is to be pulverized, *F* is removed, and the substance is placed in the hollow cylinder, *DE*; the cylinder *F* is replaced, and struck forcibly with a mallet. When the two cylinders are separated, the substance will be found reduced to a rough powder. If necessary, this powder can be brought to an impalpable state in an agate mortar.

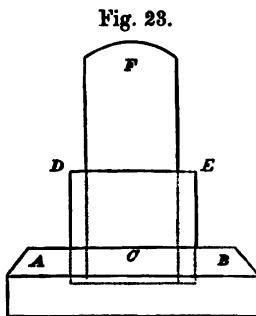


Fig. 23.

8. CHARCOAL BORERS.—For quantitative Blowpipe analysis, grooves of various dimensions are bored in the charcoal that serves as a support. There are three different borers, of hardened steel, which will be here described :—

a. A borer, of the form of Fig. 24, for making a cylindrical cavity, in which silver, gold, or copper may be heated. It is a four-sided pyramid, the sides being ground from below with a three-edged file, so that it appears like a double chisel, crossing at a right angle. Its under diameter is one-third of an inch. This instrument may be held in a wooden case, containing a brass ring.

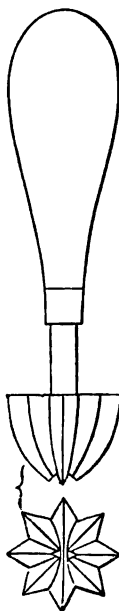
Fig. 24.



When a groove is to be made with the borer, it is placed at a

right angle, with the charcoal, pressed upon it with force, and turned upon its axis until the groove is sufficiently deep. The instrument is then withdrawn, and the charcoal-dust removed from the cavity. A silver assay should have a deeper groove than a copper one, owing to the former metal requiring, when operated upon, to be mixed with lead.

Fig. 25.



b. A second Charcoal borer, for forming larger grooves, Fig. 25. Its upper diameter is seven-eighths of an inch, and its length three-fourths of an inch. This borer is handled in the same manner as the preceding one for the formation of grooves in the charcoal.

The cavity formed by this instrument serves as a support in roasting different substances whose metallic contents are to be determined quantitatively.

c. A third Charcoal borer, similar to Fig. 26, has one end of the same form as that first described, but the double chisel is only $\frac{3}{16}$ ths of an inch in diameter, and is employed for making incisions in the charcoal, to contain quantitative lead and tin assays. The other end has a diameter of $\frac{3}{8}$ of an inch, it is sharp like a spatula, and serves to make small grooves for qualitative experiments.

Fig. 26.

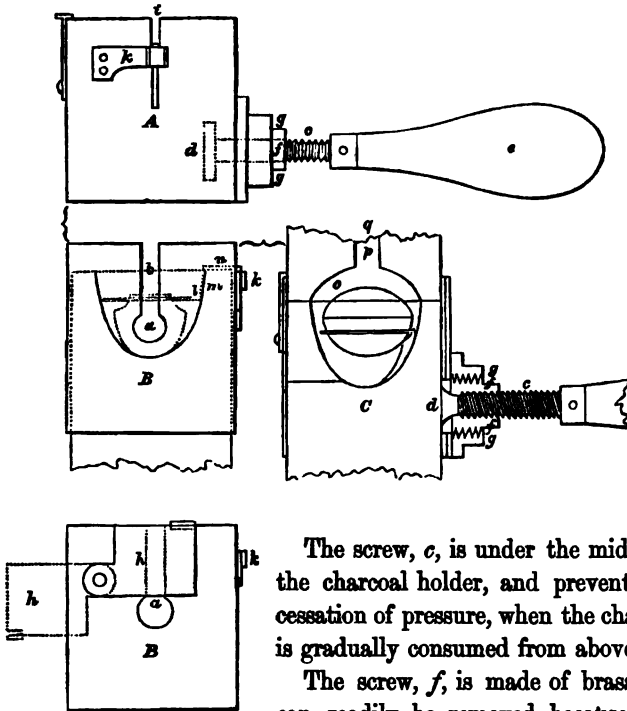


9. A CHARCOAL SAW.—This instrument should have very fine teeth, for cutting with ease long pieces of Charcoal. The most useful size is five inches in length, three-eighths of an inch in breadth, one sixteenth of an inch in thickness. It should be provided with a wooden handle, three inches in length.

10. A CHARCOAL HOLDER.—In quantitative assayings, which are to be melted without the accession of atmospheric air, the charcoal must be encased in iron foil. Fig. 27 represents several sections of this instrument. Its sides are $1\frac{3}{8}$ inch in breadth, and $1\frac{1}{2}$ inch in height. At the anterior side, B, it is provided with a

round opening, *a*, terminating at *b*; and at the reverse, is a small iron screw, *c*, provided with a wooded case, *e*, on whose inner end is an iron disc, *d*, which revolves round its axis.

Fig. 27.

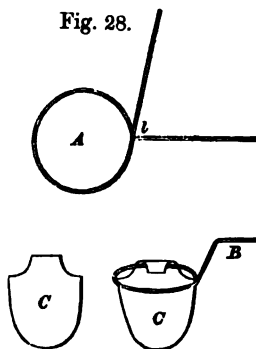


The screw, *c*, is under the middle of the charcoal holder, and prevents the cessation of pressure, when the charcoal is gradually consumed from above.

The screw, *f*, is made of brass, and can readily be removed, because it is inserted at *g*. A small iron plate, *h*, is fastened, by a rivet, to the face of the charcoal holder, so that the incision, *b*, can be opened or closed, as the figure shows. On the right side, *A*, of the instrument, is a small opening, *i*, a quarter of an inch in length, for the reception of the platinum wire, to be described hereafter; and under this aperture is a small brass case, *k*, for holding its end.

11. PLATINUM WIRE AND FOIL.—When an ore is to be roasted, or an assay melted, in a clay basin, the latter must be supported by some substance placed in the cavity of the charcoal. For this

purpose, a strong platinum wire, three inches and a half in length, will be found applicable. On the end of the platinum wire, a ring—Fig. 28—A, is made, by means of the small forceps before described, and the touching point, *l*, is curved to an obtuse angle: then, into the charcoal holder previously described, a charcoal prism, of a corresponding size, is inserted so that its upper side nearly reaches the cavity, *i*, of the instrument, Fig. 27. When this is accomplished, a depression is made on its surface, and the platinum wire is sunk so accurately into it that the charcoal appears to touch it uniformly in all parts. The straight part of the wire is now bent so as to follow the lines *m n k*—Fig. 27. To the curved part of the wire, a hollow thin platinum foil, *c*, Fig. 28, 9-16ths of an inch in breadth, is united, as depicted in the figure.



12. A CAPSULE OF BRASS FOIL, with the interior polished, two inches and a half in length, and of the form of Fig. 29. It is used for mixing silver and gold ores with proof lead and calcined borax; and for pouring these, or similar mingled ingredients, into a cornet of soda paper.

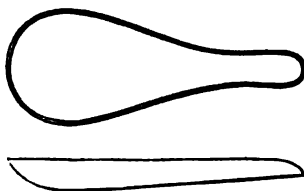


Fig. 29.

13. A SMALL SCISSORS, sufficiently strong for cutting metallic laminae, as well as soft materials.

14. A KNIFE.—For many researches, a steel knife, well sharpened and magnetized, is requisite. It is employed for mixing the various fluxes in quantitative assays, and also for determining the degree of hardness of many metallic bodies.

15. Some FILES, of different sizes and fineness, three-edged, flat, half-round, and round, the description of which is unnecessary, are required in many examinations.

16. AN IRON INSTRUMENT *for the formation of CUPELS* ; with a Bolt and Stand.—Small Cupels of bone-ashes, are necessary for refining plumbiferous alloys obtained in quantitative assayings, containing Silver or Gold. Cupellation is resorted to for the purpose of separating the noble metals as they are called, from those which are readily convertible into oxides. The operation consists in fusing the alloy on charcoal with pure lead, and then heating the resulting bead in the oxidating flame, upon a substance sufficiently porous to absorb the fused oxides produced by the ignition. The best method for the preparation of these Cupels is to cast them in a metallic mould ; in which they are to remain till the cupellation is completed.

Generally, two Moulds are employed, with concave depressions of different sizes, for the formation of cupels in large and small operations. Fig. 30, A, B, presents these moulds, as well as their bolts, *a*, *b*.

The Moulds are of iron, and, in the upper part, are 13-16ths of an inch in diameter ; the bolts are of hardened steel, partly ground, and of a circular form. In the under part of each mould, cruciform grooves are usually filed, so that when one arm of a pincers is inserted, and the other pressed against the upper part of the mould, it may easily be removed, even when heated to redness.

Fig. 31 is a small wooden stand, 3½ inches in height, having on the under part a round piece of wood, serving as a durable support. In the upper part is a strong brass wire, fastened by a screw ; and at the end of this wire is a brass cross, the arms elevated to an angle of 85°, so as to allow the mould to be conveniently placed between them. When the transverse incisions in the bottom of the iron instrument are not brought into contact with the arms of

Fig. 30.

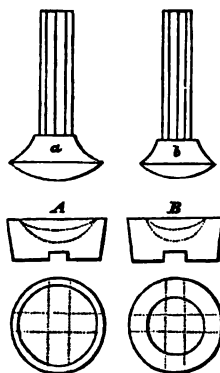
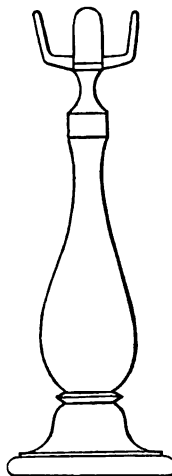


Fig. 31.



the brass cross, an operator is enabled to raise or depress the instrument at pleasure, so as to afford the required heat.

17. AN IVORY SPOON, well polished and ground, Fig. 32. 5-16ths of an inch in breadth, and of the form given in Fig. 32. The handle of this spoon, which is in the form of a spatula, is for mixing in the capsule ores with pure lead and calcined borax ; and the spoon part is necessary in the weighing of ores and such substances as are to be fused.

18. A SMALL BRUSH.—For quantitative examinations a painter's brush is indispensable, for detaching from the pans of the balance any remaining powder, *et cetera*.

19. A LEAD SIEVE.—In Blowpipe analysis, the Lead must be in a very minute state of division, to allow of its being intimately mixed with the various substances

to be operated upon. It may be obtained in this state by agitating granulated lead in a small brass sieve—Fig. 33. The method of assaying is so very delicate, that it almost always produces a bead of silver, when the common lead of commerce is submitted to trial. It is for this reason that so much care must be taken in preparing lead for

Fig. 33.



cupellation. The best method for obtaining it pure, and in a very minute state of division, is to precipitate it from a solution of the acetate, with a plate of zinc.

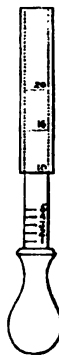
20. A STEEL MAGNET.—A strongly magnetized steel blade, or a steel magnet, about three inches and a half long, and $\frac{1}{4}$ of an inch in diameter, will be useful in ascertaining whether small particles of the ores under examination are magnetic. If, to a mixture of finely divided iron and tin, immersed in water, the end of the magnet be applied, the particles of iron will be found adhering when it is withdrawn.

21. A LEAD MEASURE.—The instrument—Fig. 34—which is similar to a gunpowder-measure, is for avoiding the weighing of a



certain portion of lead, in quantitative assayings. It consists of a glass tube, open at the extremities, containing a small wooden cylinder. This glass tube is divided into parts of equal degrees, 10, 15, 20, each of which are capable of holding 100 milligrammes of granulated lead. For quantities less than 100 milligrammes, the piston is graduated as in the figure, with

1,	2,	3,	4,	5,	corresponding to—
10,	20,	30,	40,	50	milligrammes	



22. A SMALL WOODEN CYLINDER.—a cylinder of wood is employed to make small cornets from the soda-paper described at page 19. It should be about 9-32nds of an inch in thickness, as in Fig. 35, B ; and, for convenience, should have a handle at one end.

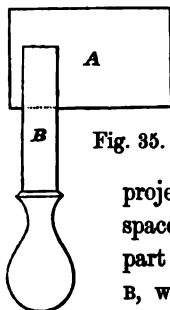
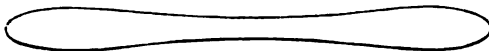


Fig. 35.

The cornet is made in the following manner:—A slip of paper, A, is so placed under the cylinder that one of its long sides projects beyond the upper end of B, as in the figure, a space equal to the diameter of the cylinder. The part of the paper so protruding is then folded back on B, with the aid of a small spoon ; the part of the paper to the left of B is next folded over the cylinder, turned in such a manner that the remaining portion of the paper to the right of B, is folded over the latter, and the closed end struck upon a hard even surface, in order that the cornet may retain its shape, after the removal of the cylinder.

23. A POLISHED IRON SPATULA, four inches in length, and of the form of Fig. 36, is employed in levigating such alligations as are to be roasted in quantitative determinations.

Fig. 36.



24. TEST-TUBES, with a Funnel and Stand.—When compounds, which contain several heterogeneous ingredients, cannot be accurately recognised in the Blowpipe flame, an operator must resort

to liquid testing, to ascertain each constituent, either qualitatively, or quantitatively. This is effected by means of test-tubes, Fig. 37, five and a half inches long, and five-eighths of an

Fig. 37.

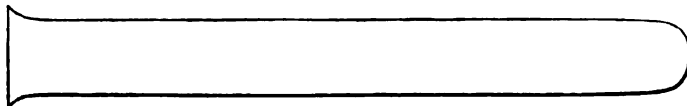
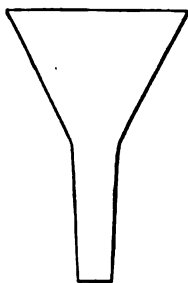


Fig. 38.



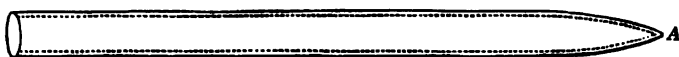
inch in diameter, which are very well adapted for containing the dissolved compound, when it is to be operated upon by reagents. They must be blown remarkably thin, so as not to crack when held in the flame of a spirit-lamp. They should also be provided with a small wooden perforated frame, serving as a support.

The Funnel, Fig. 38, is for the reception of a paper filter, into which the turbid liquid is poured from a test-tube, to separate the precipitate produced by a reagent, from the

clear solution.

25. A small Glass PIPETTE.—This instrument should be five inches in length, and three eighths of an inch in diameter, Fig. 39. It is exceedingly convenient for affusing precipitates

Fig. 39.



with hot water ; and also for withdrawing the supernatant liquor from the precipitate, in quantitative Gold and Tin analysis. The point only should be slightly contracted, and not drawn out into a capillary tube.

26. AN EDULCORATOR.—The Edulcorator, or, as it is generally termed, the wash-bottle,—Fig. 40,—is a German glass flask, capable of holding about a pint of water, the mouth of which is

closed by a cork ; through this two bent tubes are passed, the one, *a*, for the admission of air into the bottle, and the other, *b*, which reaches nearly to the bottom, for the escape of the water.

Fig. 40.



The external orifice of the latter is drawn to a fine point, which should never exceed one-fortieth of an inch in diameter. The opening of the tube can be widened by grinding it upon a sandstone, or rubbing it with a file which has been moistened with camphorized turpentine ; or it may be narrowed by holding it in the flame of the spirit-lamp, or before the Blowpipe. The bottle, when corked, should be a little more than half full.

When wanted for use, the operator blows into the tube, *a*, and the pressure to which the water is subjected, forces it through the tube in a fine stream, which can be directed upon the precipitate in the filter.

Near the end of the edulcoration, the jet of water should be directed towards the edges of the filter, and not upon the precipitate itself, by which means any particles of the latter adhering to the paper, are washed to the bottom, and brought into a small compass.

Gelatinous precipitates require much washing ; the jet of water may occasionally be deficient in force, and not strong enough to detach them. In this case they may be stirred up with a glass rod, but the operator must be careful not to force the rod through the paper, otherwise the filtration will have to be repeated. It is to be observed, that after every addition of water, no more is to be added until the first quantity has filtered through, otherwise an affusion of the precipitate is not effected, but only a continued dilution of the solution.

27. WATCH-GLASSES, two inches in diameter, Fig. 41, *a*.—

D

These may be employed either as test-glasses, or as crucible-covers during the refining process.

28. GLAZED PORCELAIN BASINS, of various sizes, are very useful for the precipitation or evaporation of liquids and also for heating, in the dry state, earths and metallic oxides with reagents. The form of two such vessels is given. in Figs. 41 and 42.

29. A cylindrical BOTTLE, of lacquered sheet iron, provided with a screw top, for containing lamp oil.

Fig. 41.

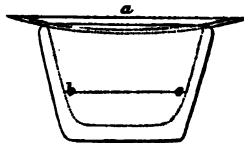


Fig. 42.



VI.—REAGENTS.

Those substances which operate upon others in any remarkable manner so as to lead to their detection, whether by change of color, precipitation, effervescence, or other characteristics, are called reagents.

They are divided into *general* and *special* reagents; those denominated general, are employed to determine the *class or group* to which the substance under examination belongs; those termed special, are used for the detection of individual bodies. For the sake of clearness and simplicity, these two classes are subdivided as follows :—

REAGENTS IN THE HUMID WAY.

I.—GENERAL REAGENTS.

- a.*—Reagents used as simple solvents.
- b.*—Reagents used as chemical solvents.
- c.*—Reagents used to separate, or otherwise characterize, groups of substances.

II.—SPECIAL REAGENTS.

- a.*—Reagents used for the detection of bases.
- b.*—Reagents employed for determining the presence of acids.

REAGENTS IN THE DRY WAY.

FLUXES AND BLOWPIPE REAGENTS.

Each reagent will now be considered according to the plan thus laid down.

REAGENTS IN THE HUMID WAY.

I. GENERAL REAGENTS.

a. Reagents used as simple solvents.

1. DISTILLED WATER. HO.

Uses.—Principally as a simple solvent for a great variety of substances. It also effects the transformation of several neutral metallic salts into soluble acid and insoluble basic compounds; this is particularly the case with antimony and bismuth, which are precipitated in the form of a white powder, on the addition of *much* water. Tartaric acid readily dissolves or prevents the formation of the basic salt of antimony, which is thus easily distinguished from bismuth.

Tests.—It should not leave the slightest residue upon evaporation; neither should it alter the color of blue or red litmus, or any other test-paper. Nitrate of silver, chloride of barium, oxalate of ammonia, or lime-water must not cause the least turbidity.

2. ALCOHOL. $C^4H^6O^2 = AeO, HO.$

There are two kinds; absolute alcohol, which, at 60° Fahr., should have a specific gravity of .796 or .797, and spirit of wine having a density of from .830 to .840 at the same temperature.

Uses.—It is frequently employed for separating the substances soluble from those which are insoluble in it; as, for instance, chlorides of barium and strontium, the latter being dissolved by this menstruum.

It serves likewise to precipitate bases insoluble in it from their aqueous solutions, *exempli gratia*, malate of lime.

Alcohol is used also for the production of acetic and other ethers; to co-operate with acids in the reduction of chromic acid, peroxide of lead, *et cetera*; for detecting various substances

which impart a characteristic tint to the flame, especially boracic acid and strontia, and for detecting potassa combined with silicic acid, which, before the Blowpipe alone, gives the same reaction as soda or lithia.

Tests.—Pure alcohol will completely volatilize, and neither causes any smell of fusel oil, when rubbed between the hands, nor reddens blue litmus paper.

3. ETHER. $C^4H^6O = Ae\ O$.

Ether is little used for the analysis of inorganic bodies. It is principally employed for dissolving bromine, when isolated by other reagents, and for this purpose the commercial article is sufficiently strong and pure.

b. Reagents principally employed as chemical solvents.

1. HYDROCHLORIC ACID. $H\ Cl$.

Uses.—It is very extensively used as a solvent, and for the detection of silver, suboxide of mercury, lead, and ammonia.

From solutions of the three former, it precipitates chloride of silver, subchloride of mercury, and chloride of lead, all of which are white. The chloride of silver is insoluble in nitric acid, but easily soluble in ammonia; the subchloride of mercury blackens on the addition of ammonia; and the chloride of lead is soluble in a large proportion of water.

When a glass rod moistened with dilute hydrochloric acid is brought into close contiguity with a solution containing free ammonia, white dense clouds of chloride of ammonium are formed.

Tests.—When intended for analysis, it must be colorless, and leave no residue upon evaporation. Chloride of barium should produce no precipitate, neither in the highly diluted acid, nor after ebullition with nitric acid. Sulphide of hydrogen must leave it unaltered. After neutralizing with ammonia, and acetic acid has been added in slight excess, ferrocyanide of potassium must not produce any precipitate, nor even impart to it the slightest tinge of blue.

2. NITRIC ACID. H O, N O^5 .

Uses.—Nitric acid is employed as a solvent for metals, oxides, sulphides, *et cetera*. Metals, or sulphides, in dissolving, are first converted into oxides, and subsequently into nitrates. This acid dissolves most oxides, forming with them nitrates; the same occurs with most combinations of insoluble salts, the weaker acids being expelled by the nitric acid in the process of solution.

Nitric acid is used frequently as an oxidising agent, as to convert the oxide of iron into the sesquioxide, *et cetera*.

Tests.—When pure, it is colorless, and leaves no residue when evaporated to dryness on platinum foil. Chloride of barium, or nitrate of silver must not in the least impair its clearness. Before adding these reagents, the acid should be slightly diluted, otherwise nitrates will be precipitated.

3. NITRO-HYDROCHLORIC ACID, AQUA REGIA. $\text{N O}^2 \text{Cl}^3$.

Is generally prepared when wanted, by adding one part of pure nitric acid to two of hydrochloric acid. When kept for any length of time it decomposes.

Uses.—Principally as a solvent for gold and platinum.

4. ACETIC ACID. $\text{C}^4 \text{H}^4 \text{O}^4 = \bar{\text{A}}$.

Uses.—This acid possesses a greater solvent power for certain substances than others, and is accordingly used to separate the former from the latter, as, for distinguishing oxalate of lime from phosphate of lime, the latter being easily dissolved, while the former remains unaltered. This reagent is also used for acidulation when it is necessary to avoid the presence of mineral acids.

Tests.—It must leave no residue when evaporated upon platinum foil. When diluted, sulphide of hydrogen should cause no precipitate; nitrate of silver or chloride of barium should not render it turbid. Should the latter reagent cause a precipitate when the acetic acid has been boiled with pure nitric acid, sulphurous acid is present.

5. CHLORIDE OF AMMONIUM. NH_4Cl .

Uses.—For retaining certain salts and oxides in solution, when others are precipitated by ammonia or some such reagent, as magnesia, which is held in solution by ammonia and chloride of ammonium, whilst baryta, strontia, and calcia are precipitated by carbonate of ammonia; it is afterwards thrown down by phosphate of soda; this reagent is also used for precipitating from potassa solutions substances which are insoluble in ammonia and its salts; *exempli gratia*, alumina, which—in the separation of the third, or sulphide of ammonium group—after precipitation of sesquioxide of chromium by boiling, is thrown down by chloride of ammonium.

Tests.—When heated upon platinum foil it must completely volatilize. It must not be affected by sulphide of ammonium, and its reaction on test-paper should be perfectly neutral.

c. Reagents used to separate, or otherwise characterize groups of substances.

1. REAGENT PAPERS.

a. Blue litmus paper.—This is prepared by immersing slips of fine unsized paper in a decoction of litmus, for some time. When thoroughly saturated, they are suspended upon a thread to dry.

Uses.—For detecting free acid in solution, its color being changed to red.

b. Reddened litmus paper.—A decoction of blue litmus is treated with sulphuric acid until it assumes a red tint. The slips of paper, which are then passed through this, should be distinctly red when dry.

Uses.—For the detection of free alkali, its color being restored to blue.

c. Brazil wood paper.—If Brazil wood be boiled in water for a sufficient time, it communicates a fine red color to the fluid. It must then be filtered, and the slips of paper immersed in it, and dried as before.

Uses.—It is an excellent reagent for detecting hydrofluoric

acid, being tinged straw-yellow, when immersed in a very dilute solution of this acid.

d. Turmeric paper.—Turmeric root is first reduced to powder, and then treated with boiling alcohol; the decoction must be filtered, and the paper saturated with it.

Uses.—For detecting free alkalies: the change produced is very characteristic, its bright yellow color becoming dark brown.

2. SULPHURIC ACID. $\text{H}_2\text{O}, \text{SO}_3$.

Uses.—Sulphuric acid has a greater affinity for most bases than any other acid, and is, consequently, employed for liberating them, especially hydrochloric, nitric, acetic, boracic, and phosphoric acids from their combinations. It is used for preparing hydrogen, sulphide of hydrogen, and other gases. Many substances which cannot exist anhydrous, are decomposed when brought into contact with it, owing to its great affinity for water. It is a special test for the detection of baryta, strontia, and lead, with which it gives white precipitates; when used for this purpose it should be dilute. It also serves as a reagent for lime, when this earth is in combination with phosphoric acid, and shows no decided reaction before the Blowpipe.

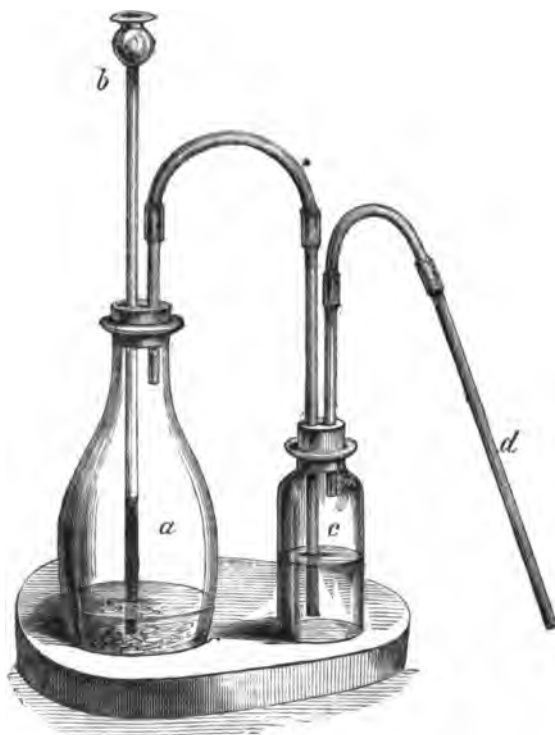
Tests.—It must not destroy the color of a solution of indigo when boiled with it. When mixed with pure zinc and water it will give hydrogen, which, on being passed through a tube heated to redness by the flame of a spirit-lamp, should not deposit arsenic.

3. SULPHIDE OF HYDROGEN. HS .

Sulphide of hydrogen gas is prepared in the following manner:—Into the flask *a*, Fig. 43, is introduced a small portion of pure sulphide of iron, and it is then tightly corked up. Water is poured in till it is covered, and concentrated sulphuric acid added through the funnel tube *b*. The small bottle *c* contains distilled water for the purification of the gas, which is finally evolved through the tube *d*, into the solution intended to be saturated.

Sulphide of hydrogen water,—hydrosulphuric acid—may be prepared by completely saturating cold distilled water with the gas.

Fig. 43.



Uses.—It is an invaluable reagent for separating metals into the principal groups. It precipitates cadmium, copper, lead, tin, mercury, antimony, arsenic, silver, gold, platinum, *et cetera*, from their acid solutions. It is frequently employed for reducing the oxides of metals as well at the ordinary as at a high heat, and also highly oxidized bodies to a lower degree of oxidation.

Tests.—Sulphide of hydrogen water should be quite clear, possessing the odor of the gas in an eminent degree: addition of ammonia must not cause a black coloration: when treated with sesquichloride of iron it should yield a large precipitate of sulphur.

4. SULPHIDE OF AMMONIUM. NH_4S .

Uses.—This reagent is of great value, and divides those metals which are precipitated by sulphide of hydrogen as sulphides, into two groups, a few of them being soluble, while the remainder is insoluble in it. It is also the precipitant of the third group; alumina and sesquioxide of chromium being thrown down as bases, and nickel, cobalt, manganese, iron, and zinc, as sulphides. It also precipitates baryta, strontia, and lime, when in combination with phosphoric, oxalic, and boracic acids, and magnesia when united to phosphoric acid.

Tests.—Sulphide of ammonium must be transparent, must completely volatilize when heated upon platinum foil, and ought not to precipitate sulphate of magnesia.

5. SOLUTION OF POTASSA. K O , H O .

Uses.—As a precipitant for many oxides it is invaluable. Some of these dissolve in excess, as alumina, oxide of chromium; while others, as sesquioxide of iron, *et cetera*, remain undissolved. In consequence of this property, it is employed to separate the former from the latter. Potassa has a great solvent power for many salts, such as sulphides and antimonie acid. Many oxides precipitated by potassa have a peculiar hue, or possess other characteristics by which they are easily recognised. Potassa expels ammonia from its salts, when heated in contact with them, and may be detected by its odor.

Tests.—Solution of potassa must be colorless, and after neutralizing it with pure nitric acid, which should occasion no effervescence, it must yield no precipitate, neither with chloride of barium nor nitrate of silver. It ought to retain its transparency when heated with an equal portion of solution of chloride of ammonium. When evaporated to dryness, and redissolved in water, no residue of silicic acid should remain.

6. AMMONIA. NH_3 .

Uses.—For neutralizing acids, and precipitating many oxides, some of which redissolve in excess of the reagent, whilst others

are insoluble in free ammonia, thus affording a ready means of separating them from each other.

Tests.—Ammonia must be colorless, must not leave any residue when evaporated on platinum foil, nor should lime-water impart turbidity in the slightest degree. Neither chloride of barium nor nitrate of silver must cause the smallest precipitate after supersaturation with pure nitric acid.

7. CARBONATE OF AMMONIA. $\text{NH}_4\text{O}, \text{CO}_2$.

Uses.—As a precipitant of the alkaline earths, baryta, strontia, calcia, and magnesia, it throws down many metallic oxides, some of which are soluble, and others insoluble. Complete precipitation of many of them only occurs on boiling. It is also employed for separating glucina from alumina, and the oxide of uranium from sesquioxide of iron.

Tests.—It must completely volatilize. Neither chloride of barium, nitrate of silver, nor sulphide of hydrogen should color or precipitate it, after complete neutralization with pure nitric acid.

8. CHLORIDE OF BARIUM. Ba Cl .

Uses.—From its properties of forming soluble salts with certain acids, and insoluble with others, it is an invaluable reagent for subdividing the precipitable acids. As a special reagent for sulphuric acid, with which it gives a white precipitate insoluble in all acids, its worth cannot be estimated.

Tests.—The solution of chloride of barium must not be colored, or rendered turbid by sulphides of hydrogen, or ammonium. The filtrate, after adding an excess of pure sulphuric acid, ought to leave no residue when evaporated to dryness on platinum foil.

9. NITRATE OF BARYTA. $\text{Ba O}, \text{N O}_5$.

Uses.—The same as chloride of barium, and is substituted for that reagent where the presence of a metallic chloride would be deleterious.

Tests.—The same as those for chloride of barium, except that nitrate of silver must produce no precipitate.

10. CHLORIDE OF CALCIUM. Ca Cl .

Uses.—It precipitates, as a group, from neutral solutions, phosphoric, hydrofluoric, oxalic, tartaric, and citric acids ; also, if the solution is not very dilute, sulphuric acid ; better, however, in the presence of alcohol. The different aspects and characteristics of the precipitated lime salts, as to their solubility and insolubility, *et cetera*, enable the analyst to distinguish certain individual acids—see Lime-Water, page 47.

Tests.—Solution of chloride of calcium must be neutral, and ought not to be rendered turbid or discolored by sulphide of ammonium.

11. NITRATE OF SILVER. Ag O, NO^5 .

Uses.—It is a very important reagent for the classification of acids into groups, and is employed for the detection of certain individual acids, especially hydrochloric, phosphoric, and arsenic acids. With the first, it gives a white precipitate insoluble in nitric acid, but soluble in ammonia ; from solutions of tribasic phosphates it precipitates lemon-yellow phosphate of silver, soluble, with difficulty, however, in acetic acid ; with arsenic acid it produces a reddish-brown precipitate.

Tests.—After adding an excess of pure hydrochloric acid, the filtrate must leave no residue upon evaporation, and should neither be colored nor precipitated by sulphide of hydrogen.

12. SESQUICHLORIDE OF IRON. $\text{Fe}^3 \text{Cl}^3$.

Uses.—As the reagent for a group of organic acids, with which, *videlicet*, succinic, benzoic, hydrosulphocyanic, acetic, and formic acids, it gives very distinct and characteristic reactions. With succinic acid it gives a voluminous brownish-red precipitate, readily soluble in acids, and which is rendered darker on addition of ammonia, owing to the withdrawal of a quantity of succinic acid. The precipitate from benzoic acid is pale-yellow, and comport itself as succinic acid. To solutions of acetates, sulphocyanides, and formiates, it imparts a blood-red color. It is employed also for the detection of hydroferrocyanic acid, in the formation of Prussian blue.

II.—SPECIAL REAGENTS.

*a. Reagents used for the detection of bases.*1. SULPHATE OF POTASSA. K O, S O_3 .

Uses.—It precipitates from solutions of salts of baryta and strontia the insoluble sulphates of the oxides. It also produces a precipitate in concentrated solutions of lime, but only after some time.

2. CHROMATE OF POTASSA. K O, Cr O_3 .

Uses.—It precipitates, from solutions of the salts of many metallic oxides, chromates; most of them are very difficultly soluble, and possess characteristic colors, by which the particular metal may often be with certainty detected. It is used principally, however, as a test for lead, with which it gives a yellow precipitate.

3. CYANIDE OF POTASSIUM. K Cy .

Uses.—It is a valuable reducing agent, and also precipitates many of the heavier metals; with nickel it gives a greenish-white, with cobalt a brownish-white, with copper a yellowish-green, and with protoxide and sesquioxide of iron a reddish-brown precipitate. The latter, on addition of an excess of hydrochloric acid, and application of heat, dissolves, giving a beautiful blue liquid.

Tests.—The precipitate produced by lead-salts must be perfectly white; the residue left upon evaporating its solution, previously saturated with pure hydrochloric acid, must clearly and perfectly dissolve in distilled water.

4. FERROCYANIDE OF POTASSIUM. $\text{K}^2 \text{Cfy}$.

Uses.—Principally for the detection of oxide of copper, with which it gives a purple-red precipitate; and for indicating the presence of sesquioxide of iron, from the solutions of which it throws down a beautiful blue precipitate. With salts of the protoxide of iron, absolutely free from sesquioxide, it gives a white precipitate, which, on exposure to the air, becomes blue.

5. FERRICYANIDE OF POTASSIUM. K^3Cfy^2 .

Uses.—As a test for protoxide of iron and its salts, with which it immediately gives a blue precipitate of ferricyanide of iron. Fe^3Cfy^2 .

6. SULPHOCYANIDE OF POTASSIUM. K^2Csy .

Uses.—The same as those of ferrocyanide of potassium for the detection of sesquioxide of iron, the most minute trace of which, in a hydrochloric acid solution, gives with this reagent, a blood-red color.

7. PHOSPHATE OF SODA. $2NaO, HO, PO^5$.

Uses.—Phosphate of soda precipitates all the alkaline earths, but is employed, after the separation of baryta, strontia, and lime, for the detection of magnesia, which it precipitates even in the presence of an excess of ammonia or its salts.

Tests.—It must not become turbid when heated with ammonia. Dilute nitric acid must completely dissolve the precipitates produced in it by nitrate of silver and chloride of barium.

8. OXALATE OF AMMONIA. NH^4O, \bar{O} .

Uses.—It is an invaluable test for lime, with which it gives a white precipitate, insoluble in acetic, but readily soluble in hydrochloric or nitric acids. This reagent produces a similar precipitate with baryta and strontia; the manipulator must, therefore, have positive proof of the absence of those earths before testing for lime.

9. PROTOCHLORIDE OF TIN. $SnCl$.

Uses.—It is a very powerful reducing agent, and is employed as a test for mercury, throwing it down as a grey precipitate, which, on rubbing, collects into globules of the metal. It is also used as a test for gold, in the solutions of which it produces a purple color or a similarly colored precipitate.

10. BICHLORIDE OF PLATINUM. $PtCl^2$.

Uses.—For the detection of ammonia and potassa, from solu-

tions of which, especially on addition of a little hydrochloric acid and alcohol, it throws down yellow crystalline precipitates, double chlorides of platinum and ammonium, or potassium as the case may be,— NH^4Cl , PtCl_2 , or KCl , PtCl_2 . When testing for potassa, the student must be sure that ammonia is absent, by having previously heated the substance to redness.

11. TERCHLORIDE OF GOLD. AuCl_3 .

Uses.—As a test for the protosalts of tin, with which it gives a purple color, or a similarly colored precipitate.

12. ZINC. Zn .

Uses.—Metallic zinc reduces many metallic salts and oxides causing their precipitation; this is owing to its affinity for oxygen. It is principally employed, however, for the reduction of antimony and tin, and, when mixed with sulphuric acid, for the evolution of hydrogen gas.

13. COPPER. Cu .

Uses.—Copper turnings are used for the reduction of mercurial salts, and for the detection of arsenious acid; when immersed in a solution of the latter, they immediately become covered with a grey metallic coating, which, on heating, separates in black scales. Copper also indicates the presence of nitric acid, when heated with substances containing it, to which dilute sulphuric acid has been added, by giving off fumes of binoxide of nitrogen; these, on being liberated, absorb oxygen from the atmosphere, and assume a brownish-red color, having the composition N O^2 .

14. IRON. Fe .

Uses.—Iron, like zinc, reduces and precipitates many metallic salts and oxides. It is employed, in the form of clean wire, for indicating the presence of copper, which falls upon it, when immersed in the solution, in a film, of the usual color of that metal. It is also used in quantitative cupellations, and to reduce phosphates to the state of phosphides. In this process, a white, brittle, metallic bead is formed, which will be noticed under phosphoric acid.

b. Special reagents employed for determining the presence of acids.

1. ACETATE OF POTASSA. $\text{K O}, \bar{\text{A}}$.

Uses.—For precipitating phosphate of sesquioxide of iron from hydrochloric acid solutions of phosphates of the alkaline earths, and for precipitating from simple solutions in mineral acids substances insoluble in acetic acid. It is also employed for recognising the presence of tartaric acid, giving a white crystalline precipitate, which, however, from the time occupied in its formation, and its great solubility, is not very characteristic.

2. HYDRATE OF LIME. LIME-WATER. $\text{Ca O}, \text{H O}$.

Uses.—The former,—hydrate of lime,—is employed for the liberation of ammonia, and the latter,—which should be kept from the air as much as possible, on account of the formation of carbonate of lime—for the detection of carbonic acid, with which it gives a white precipitate, soluble, *with effervescence*, in hydrochloric acid; oxalic acid, with which a white precipitate is formed, insoluble in acetic acid, but readily dissolved in hydrochloric and nitric acids; tartaric acid, from solutions of which appears, on addition of the reagent, a white precipitate dissolving easily in ammonia and cold potassa; the solution in the latter, on boiling, forms a gelatinous mass, which disappears on cooling; lastly, for indicating the presence of citric acid, with which it gives a white precipitate *on boiling*; this is soluble in ammonia and its salts, but not in potassa.

3. SULPHATE OF LIME. $\text{Ca O}, \text{S O}_3$.

Uses.—For distinguishing and separating baryta and strontia, producing an immediate precipitate with the former, but only doing so with the latter after a length of time.

4. SULPHATE OF MAGNESIA. $\text{Mg O}, \text{S O}_3$.

Uses.—For the detection of phosphoric acid, which is precipitated by it, even in the presence of ammonia and ammoniacal salts.

5. CHLORIDE OF MAGNESIUM. Mg Cl .

Uses.—The same as those of sulphate of magnesia.

6. SULPHATE OF IRON. Fe O, S O^3 .

Uses.—For indicating the presence of nitric acid ; for this purpose a clean crystal is dropped into the solution under examination, and a little dilute sulphuric acid added ; if nitric acid is present, the crystal immediately becomes coated with a brown areola. This reagent is also employed in solution for detecting hydroferricyanic acid, with which it gives a blue precipitate of ferricyanide of iron ; when mixed with perchloride of iron and potassa as a test for hydrocyanic acid. If the precipitate thus produced dissolves with a fine blue color in hydrochloric acid, the acid in question is present. Sulphate of iron is likewise employed for reducing the salts of gold.

7. NEUTRAL ACETATE OF LEAD. Pb O, A .

Uses.—It is specially applied as a test for chromic acid and soluble chromates, producing a lemon-yellow precipitate of chromate of lead. When a piece of bibulous paper saturated with this reagent is held for a short time over a solution containing sulphide of hydrogen, it becomes of a dark-brown color, and is of great use for this purpose. Acetate of lead likewise produces precipitates with some other acids, but the principal ones are here given.

8. SULPHATE OF COPPER. Cu O, S O^3 .

Uses.—As a test for arsenious acid, producing a precipitate of yellowish-green arsenite of copper ; for arsenic acid, with which it gives a greenish-blue precipitate ; when associated with caustic potassa, for distinguishing the former from the latter, arsenious acid and arsenites giving a red precipitate of suboxide of copper ; when united with sulphate of iron, for detecting hydriodic acid, throwing down white subiodide of copper, $\text{Cu}^2 \text{I}$; and, also, for indicating the presence of hydroferrocyanic acid, with which it gives a dark purple-red precipitate of ferrocyanide of copper, $\text{Cu}^2 \text{Cfy}$.

9. SUBNITRATE OF MERCURY. $\text{Hg}^2\text{O}, \text{N O}^5$.

Uses.—This reagent acts analogous with, and, in many cases may be substituted for, nitrate of silver. It is employed as a test for many acids, especially the hydracids; and also for the detection of substances easily capable of oxidation, as formic acid, which deoxidizes it, being converted into carbonic acid and water, with reduction and precipitation of metallic mercury.

10. OXIDE OF MERCURY. Hg O .

Uses.—As a test for hydrocyanic acid, dissolving only in alkaline liquids when that acid is present.

11. CHLORIDE OF MERCURY. Hg Cl .

Uses.—As a test for hydriodic acid, with which it produces a beautiful red precipitate of iodide of mercury, Hg I . On addition of this reagent to formic acid, it at first falls down as chloride of mercury, but is soon reduced to the metallic state. It serves as an oxidizing agent for chloride of tin, and other substances.

12. SULPHUROUS ACID. S O^2 .

Uses.—As a very powerful reducing agent. It precipitates metallic mercury from solutions of mercurial salts; reduces chromic acid to oxide of chromium, and is employed for the conversion of arsenic into arsenious acid. For these, and many similar purposes, it may either be used in a state of solution, or may be generated when wanted for use by adding sulphuric acid to strips of metallic copper in a flask, and applying heat.

13. CHLORINE. Cl .

Uses.—In solution, for isolating iodine and bromine. It is also employed to decompose many organic substances; it effects this sometimes by withdrawing the hydrogen, forming with it hydrochloric acid, whilst the liberated oxygen enters into a simpler but more permanent combination with the other constituents. The chlorine is best obtained by heating one part of coarsely powdered binoxide of manganese with four or five parts of common hydrochloric acid.

14. SULPHINDIGOTIC ACID,

Is a solution of one part of indigo in seven parts of concentrated sulphuric acid.

Uses.—As a test for nitric acid, which destroys its color. When the nitric acid is not in a free state, it must be liberated by adding sulphuric acid to the compound.

15. STARCH PASTE,

Is made by triturating starch with water till perfectly mixed. It should have the consistency of cream.

Uses.—When paper moistened with starch-paste is exposed to the vapors of iodine—liberated by chlorine or strong sulphuric acid—it is immediately tinged violet. When bromides are treated in a similar manner, the paste assumes an orange-yellow hue.

REAGENTS IN THE DRY WAY.

FLUXES AND BLOWPIPE REAGENTS.

The three principal reagents, indispensable in examinations with the Blowpipe, are—

Carbonate of Soda ;
Biborate of Soda, or Borax ;
Phosphate of Soda and Ammonia, or
Microcosmic Salt.

These salts especially are to be provided in a state of purity, and will therefore be described separately.

1. CARBONATE OF SODA. Na O , C O_2 .

When a current of carbonic acid gas is passed through a saturated solution of carbonate of soda in water, bicarbonate of soda is precipitated in crystalline grains, which must be well affused with cold water, and dried. In this state it may be employed for qualitative, but when required in quantitative analysis, it must be calcined in a flat porcelain basin, to liberate the second atom of carbonic acid, and its water of crystallization.

WINKLER obtains the bicarbonate of soda free from sulphate of soda, in the following manner :—He dissolves 4 ounces of crystallized carbonate of soda in 8 ounces of distilled water, and adds to the solution $1\frac{1}{2}$ ounce of carbonate of ammonia ; then heats the mixture to 132.8° Fahr. on a water-bath. After some time, a large quantity of bicarbonate of soda precipitates, which is separated from the supernatant liquor by filtration.

To ascertain whether the carbonate of soda contains any sulphate, mix two parts with one of pure silica, and fuse this mass upon charcoal, in the reduction flame ; if the smallest quantity of sulphate of soda be present, the resulting bead will be of a deep yellow or red color, owing to the formation of sulphide of sodium.

A second method is, to heat a small quantity of carbonate of soda upon charcoal, in the reduction flame, until it is entirely absorbed. Cut out, with a small knife, this part of the charcoal, and digest it in water ; to the solution, add a small piece of bright metallic silver ; if sulphuric acid had been present, the surface of the silver would become first yellow, then brown, and finally black.

The carbonate of soda, in qualitative examinations with the Blowpipe, determines—first, whether a body fuses or is rendered soluble ;—second, the presence of silicic acid in combination.

In quantitative analysis, it serves as the means of rendering soluble, silicic acid, tungstic acid, and titanous acid combinations ; and also for accelerating the reduction of different metallic oxides. The best method of preparing a large quantity of bicarbonate of soda is to pass a stream of carbonic acid gas over an atomic mixture of crystals of soda and the dry carbonate. Great heat is evolved during the process, owing to the rapidity with which the gas is absorbed.

2. BIBORATE OF SODA. $\text{NaO}, 2\text{BO}_3$.

This salt, which is met with in commerce in large masses, must be re-crystallized, to free it from foreign matters. If the crystals yielded are pure, they will give a transparent glass when heated upon the ring of a platinum wire. In quantitative analysis, owing to the crystals occupying so much space when heated, on

account of their intumescence, the borax must be employed in a calcined state. According to BERZELIUS, almost all substances dissolve when fused with borax, and form, with its ingredients, acid and basic combinations ; for this reason it is such a valuable flux in Blowpipe experiments.

3. PHOSPHATE OF SODA AND AMMONIA. $\text{NaO}, \text{NH}^4\text{O}, \text{HO}, \text{PO}^5$.

It is best obtained by heating together, in distilled water, 100 parts of crystallized phosphate of soda, with 16 parts of chloride of ammonium. Chloride of sodium separates, and the liquid, when filtered and evaporated, affords the salt in fine crystals. When this salt is heated upon charcoal, or platinum wire, it loses its water and ammonia, and is converted into metaphosphate of soda, which, in consequence of its excess of acid, has the power of fusing almost every chemical compound.

This salt, in the liquid state, detects magnesia and the protoxide of manganese.

Besides the three reagents described, there are many others necessary to facilitate the fusion, or to effect the decomposition, of certain substances heated before the Blowpipe, and also to aid in the detection of particular elements ; these are :

4. NITRATE OF POTASSA. KO, NO^5 .

This salt is obtained in small prisms, when the saltpetre of commerce is dissolved in hot water, and the liquid allowed to cool gradually.

a. The production of colors, in beads formed with borax or microcosmic salt upon the platinum wire, can sometimes be facilitated by the addition of saltpetre. A small crystal of this salt is supported near the lamp, upon a porcelain capsule, and when the bead is removed from the flame, it is instantly pressed upon the saltpetre. The globule immediately tumefies, and the oxidized metal exhibits its color on the exterior. If the Blowpipe-flame is at all altered during this examination, the reaction will be destroyed. By this expedient, a portion of manganese, so minute as otherwise to pass unnoticed, can be readily discerned, by a beautiful emerald green color being produced.

b. Nitrate of potassa also has the power of oxidizing arsenic and chromium ; converting them into acids which combine with its base.

5. BISULPHATE OF POTASSA. $\text{K O}, \text{S O}_3, \text{H O}, \text{S O}_3$.

This compound generally presents itself when the neutral sulphate is strongly heated with oil of vitriol. If the bisulphate obtained contains too much acid, which must, if possible, be avoided, it emits thick white vapors, when heated only slightly. Bisulphate of potassa, in solution, indicates lithia, boracic acid, nitric acid, hydrofluoric acid, bromine, and iodine ; and separates baryta and strontia from other earths and metallic oxides.

6. VITRIFIED BORACIC ACID. B_3 .

This acid is generally prepared by dissolving the borax in four times its weight of water, at 100°C ; the solution is filtered while hot, and a quantity of oil of vitriol, equal to one-fourth of the weight of the borax, immediately added. The sulphuric acid unites with the soda, and forms sulphate of soda, which remains in solution ; while the boracic acid, on cooling, separates in thin shining crystalline plates. These plates are drained, and being sparingly soluble, may be washed with cold water, and, afterwards, re-dissolved in boiling water, and re-crystallized. The boracic acid still retains a small quantity of sulphuric acid, probably in a state of chemical combination, and if required of absolute purity, must be fused at a red heat in a platinum crucible, then re-dissolved and crystallized. The density of the vitrified acid is 1.83. It serves, in qualitative examinations, to detect the presence of phosphoric acid in minerals, and small portions of copper in lead alloys. In quantitative analysis, it is generally used to ascertain the quantity of copper contained in a lead ore, and also the amount of copper united with various metals.

7. NITRATE OF COBALT IN SOLUTION.

The pure metal must be dissolved in dilute nitric acid, the solution evaporated to dryness, and the residue dissolved and filtered, to separate any foreign matter.

There are a few substances not easily distinguished by simple ignition, which acquire marked characters on being moistened with a solution of nitrate of cobalt, and then heated strongly in the oxidating flame. These are,—

Alumina, acquiring a beautiful *pale blue* color,
Magnesia, a *rose-red* tint,
Zinc, a bright *green*, and
Tin, a *bluish green*.

A few drops of a solution of cobalt may be placed upon the substance by means of platinum wire, or a small pipette.

8. OXALATE OF NICKEL. $\text{NiO}, \bar{\text{O}}$.

It is obtained perfectly pure in the following manner :—Heat copper-nickel—arsenic-nickel to redness with a quantity of borax in a clay crucible, and when in a state of fusion remove the crucible from the muffle, and expose it to a free current of air. If the ore had contained iron, cobalt, lead, antimony, or sulphur, they would have been oxidized and dissolved in the borax, while the nickel would remain in combination with the arsenic. Separate the nickel compound from the dross, and examine a portion of it before the Blowpipe for cobalt. If it be not free from this metal, repeat the preceding operation with borax. When the mixture is entirely free from cobalt, heat it strongly with a quantity of carbonate of soda and nitrate of potassa ; during this process the arsenic acid combines with the soda or potassa, and the oxide of nickel remains in an uncombined state. Dissolve the soluble part of the residue in boiling water, and filter. The oxide of nickel which remains, after being well affused with water, must be dissolved in hydrochloric acid, with the aid of heat, and the nickel precipitated by oxalic acid.

If the arsenic-nickel should contain copper, it must be precipitated from an acid solution of it, by sulphide of hydrogen, previously to the heating with borax.

The oxalate of nickel is used in qualitative examinations for the detection of potassa, in a salt which also contains soda or lithia.

9. BLACK OXIDE OF COPPER. Cu O .

It may be obtained artificially, by calcining metallic copper ; by precipitating it from the salts of copper with a solution of pure potassa ; or by heating nitrate of copper to redness.

It is useful for ascertaining the presence of small quantities of chlorine, when in combination, *et cetera*.

10. SILICA, OR SILICIC ACID. Si O^2 .

This compound is obtained in a state of purity as follows :—Heat to redness in a platinum crucible, powdered rock crystal—quartz,—with a quantity of carbonate of soda or potassa ; dissolve the fused compound in water ; add to the solution a quantity of hydrochloric acid, and evaporate to dryness. A fine gritty powder remains, which, being well affused with boiling water, and then ignited, gives pure silica.

Silica, with soda, is an excellent test for the presence of sulphuric acid ; and, when in combination with borax or soda, separates tin from copper.

11. FLUOR SPAR. Ca F .

This salt must be freed from its water of crystallization. It serves, when mixed with bisulphate of potassa, to detect lithia, and boracic acid, in their various combinations. It is also a reagent for gypsum, and is an excellent flux for ores.

12. SULPHATE OF LIME. Ca O, S O^2 .—Gypsum.

It must be kept for analysis in an anhydrous state. It is easily obtained free from water, by heating to redness a quantity of it, in a powdered state, in a platinum or porcelain crucible. It occurs abundantly in Derbyshire, Yorkshire, and Saxony ; and in various forms. It can be prepared artificially, by adding sulphuric acid to any soluble salt of lime.

Anhydrous gypsum, in Blowpipe analysis, serves as a reagent for fluor spar. If small quantities of these two substances be placed slightly apart upon charcoal, and heated in the oxidating flame, both melt and run into each other, producing a colorless bead, which becomes opaque upon cooling. If an excess of gyp-

sum is present, the bead will be colorless also. According to **BERZELIUS**, the clear glass appears to be a compound of hydrofluoric and sulphuric acids with lime, which, if exposed too long to the oxidating, or too short a time to the reducing flame, is decomposed, sulphurous acid being eliminated.

Sulphate of lime, sulphate of baryta, and sulphate of strontia fuse with fluor spar ; also fluorides of barium and strontium, with gypsum, as well as with fluor spar.

13. BONE ASHES.

They are employed in the cupellation of gold and silver. **HARKORT** reduced them to many states of minute division by the processes of sifting and washing. The bones are burnt until they become perfectly white, and then freed from any carbonaceous matter that may have adhered to them. This being done, they are pulverized in a mortar, and the finer portions separated by a sieve. The remaining powder is then thrown upon a filter, and treated with boiling water, which extracts the soluble matter. The washing, which is then resorted to, is for procuring the bone-ashes of a more uniform degree of fineness. The mass from the filter is mixed with water in any cylindrical glass, allowed to settle for a few moments, and then decanted ; the coarser powder is deposited at the bottom of the vessel, while the finer passes over suspended in the water. By repeated decantations in this way, sediments are obtained of different degrees of fineness, the last, or that which remains longest floating through the liquid, being the finest. The resulting powders must be kept in separate phials. The coarser ashes are used for the cupellation of rich silver ores, and the finer, for assaying ores in which only a minute quantity of gold or silver is present.

14. PROOF LEAD. Pb.

It is employed either in a finely-divided state, or else in small fragments ; but it must be entirely free from traces of gold, silver, or copper, as it is used in the quantitative analysis of these metals. The lead from the silver-smelting furnaces will answer both purposes, if properly sifted. If such lead is not easily ob-

tained, the operator can prepare his proof lead in the following manner :—Dissolve acetate of lead in a small quantity of hot water, filter, and then add plates of zinc to the solution until the metallic lead is wholly deposited. Dry the resulting lead between folds of bibulous paper, and keep it at hand in a stoppered phial. When this lead is employed in silver and gold cupellations, it need not be accurately weighed, but quantities of it taken, to save time and trouble, according to the standard given in the lead measure, previously described at page 30.

The method just described is the best for obtaining lead perfectly pure, and in a minute state of division.

15. CHLORIDE OF SODIUM. NaCl .

It is taken either in a powdered or melted state ; but its use is very limited. It is principally employed for glazing the earthen crucibles in which quantitative lead assays are performed.

16. CHARCOAL POWDER.

This can easily be obtained by triturating pieces of charcoal in a mortar. It is generally employed for roasting or smelting, in quantitative tin or lead examinations.

17. GRAPHITE—Plumbago.

It is used quantitatively, when free from impurities, for roasting earths, minerals, and various products, upon copper. When a good kind of graphite cannot be obtained, pure anthracite may be substituted. The graphite which is generally met with is so impure, that an operator, to get the best pieces, must pick from a mass of it those fragments which are unctuous to the touch, and in scales. These parts are then heated in a crucible, and afterwards well pulverized. If this graphite contains over ten per cent. of foreign matter, it must, previously to being used, be digested in nitro-hydrochloric acid to free it from iron and other impurities. Plumbago is extensively used for the manufacture of crucibles, as they are very durable, and sustain an intense heat. The composition of the plumbago of Cornwall is 96.0 carbon, and 4.0 iron.

18. TIN. Sn.

Tin foil is generally used, cut into little slips, half an inch broad, and tightly rolled.

Tin serves for the highest degree of reduction in glass fluxes, where small quantities of oxides of the metals are present, which are capable of being reduced to a lower state of oxide, in which state the results are more convincing. The operator brings the globule, heated in the reducing flame, in contact with the free end of a rolled rod of tin. By this means a small portion of melted tin is deposited upon the flux. The whole is then fused perfectly in the reducing flame. When the tin has been added to the bead, which contains the substance for examination, an operator must not direct the flame upon the glass for too long a time, partly because the tin would become in such a state as to prevent metallic oxides, which should only be reduced to the state of protoxide, from manifesting their presence by the peculiar color imparted to the bead, or pearl, and partly because so much tin would be dissolved, particularly if phosphates were present, that the globule, upon cooling, would be quite opaque, thus rendering the analysis of no value.

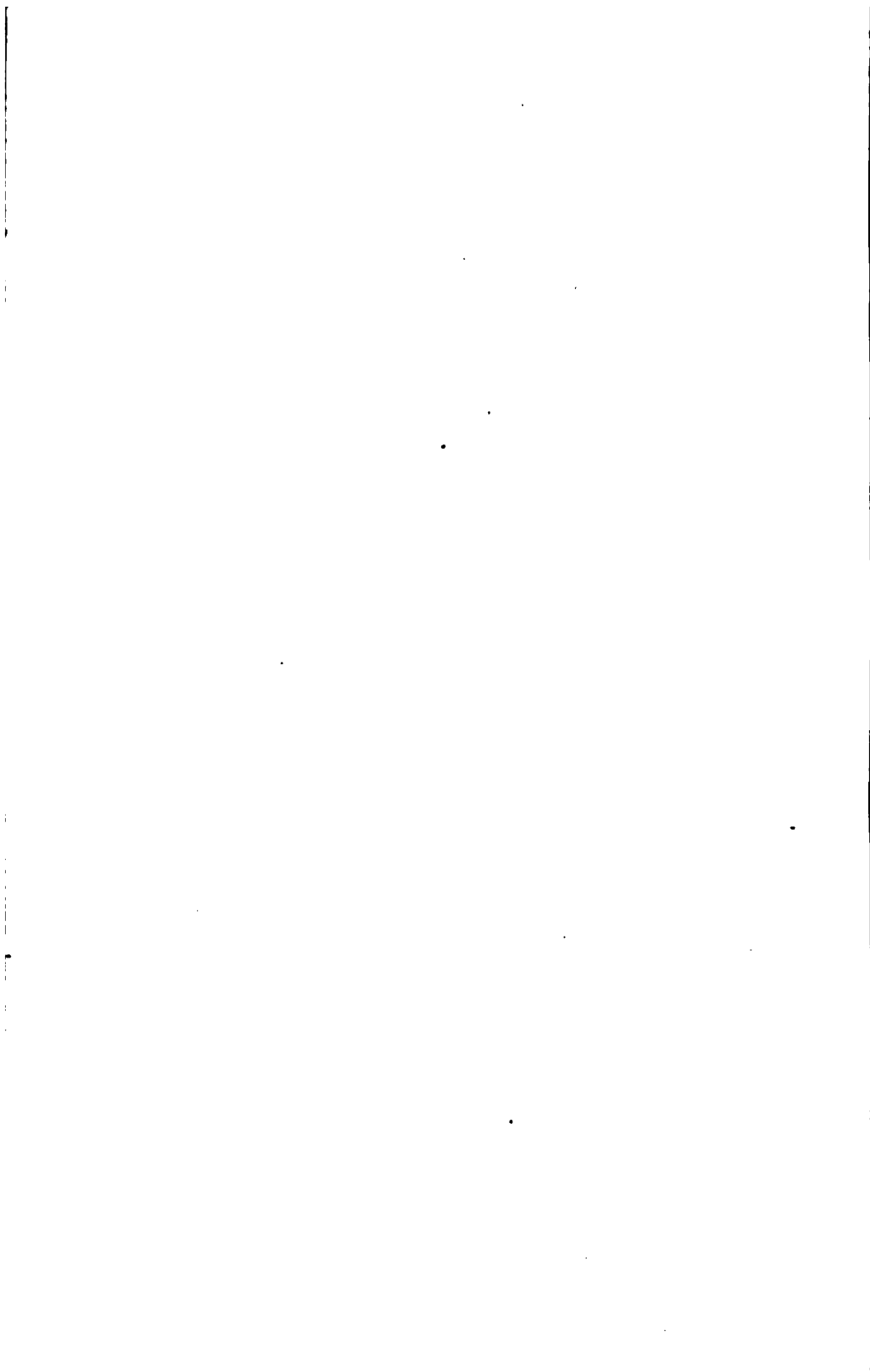
19. SILVER WIRE.

A small silver wire is useful for ascertaining the presence of hepar—sulphide of antimony,—or soluble sulphides. Silver which is obtained from the reduction of hornsilver—chloride of silver,—and beaten into wires, is also necessary in many quantitative gold examinations.

20. TARTARIC ACID. $C^8H^4O^{10} = \overline{T}$.

It is useful, with sulphide of ammonium, for separating yttria and zirconia from iron. It also detects small quantities of arsenic in metallic combinations ; and is employed in quantitative cupellations.

SECTION II.



SECTION II.

QUALITATIVE ANALYSIS

WITH THE BLOWPIPE.

It is well known that the miner frequently encounters substances, which, from their exterior appearances, he may have considered to belong to some certain class of minerals, but which, by subsequent chemical analysis, are proved to be of an entirely different constitution. It happens that in the earthy gangues of the more frequently occurring ores, minerals of great scarcity and value exist, but in such a minutely disseminated state as to be scarcely discernible with the aid of the microscope. Again, it is very difficult for the smelter to know the nature of the ingredients given him to assay. The mixed mass is given him ready for furnacing, and, except it be from previous experience, he is quite unable to judge, by its appearance the degree of heat necessary for its fusion; and difficulties may again stand in his way, with respect to the further working of his furnace products, from the presence of substances which he either does not know to be contained therein at all, or which he may consider to exist only to a very small extent. A means is wanted by which the miner and smelter can obtain the necessary information with respect to the ingredients of minerals, ores, and furnace products, and as it seldom happens that there is a laboratory within such a distance as to be available upon every emergency, the Blowpipe

affords the only resource from which this knowledge may be obtained with certainty.

By means of the Blowpipe, the substances which comprise a mixed mass can in all cases be determined. It is, therefore, my intention now to treat of the Blowpipe as the agent for Qualitative Analysis. I will describe the qualitative examination of alkalis, earths, metals, their oxides, and the non-metallic bodies, so far as they have come under my own observation ; and I shall attach to each the name, some instruction as to its nature, whence derived, and the mineralogical or chemical formula ; and I will also give general rules, in as short and concise terms as possible, with respect to the comportment of earths, metallic oxides, *et cetera*, before the Blowpipe alone, or with borax, microcosmic salt, carbonate of soda, and a solution of nitrate of cobalt ; and also a description of the different appearances presented by the sublimates of some metals, when heated upon charcoal.

I.—GENERAL RULES FOR QUALITATIVE BLOWPIPE EXAMINATIONS.

¶ *A General Rules by which the ingredients of Minerals, et cetera, may, for the most part, be detected, when heated alone or with reagents in the Blowpipe flame.*

It is always advisable, before commencing an analysis, to spread a sheet of clean glazed paper upon the table, on the middle of which the Blowpipe lamp is placed ; by this precaution, any substance falling from the charcoal or wire can be re-obtained, and the investigation continued. If it is of any consequence that the table be preserved from the hot falling assays, three or four sheets must be employed. The presence of foreign matter upon the paper must be particularly guarded against, otherwise, on a bead falling and coming in contact with it, a false result would necessarily follow. Fresh paper should be employed for each analysis.

With respect to the quantity of the substance which should be assayed when seeking its behaviour alone with the reagents before the Blowpipe, it is always advisable that too much be not taken. If, for instance, an analyst wishes to examine the solubility of an earth, mineral, *et cetera*, with borax or microcosmic salt, on the platinum wire, a piece about the size of a mustard seed is employed. In cases where metallic oxides are present, and the color only of the bead or glass is sought, considerably more is used, and in a pulverized state; but if it is the reduction of a metal by means of carbonate of soda upon charcoal, a still larger quantity is taken, and also in a powdered state. Experience, however, soon teaches the proper proportions in the different cases occurring. *If too much substance be employed*, it inevitably follows that uncertain results are the consequence.

In examining the relation of a substance by itself, or with fluxes before the Blowpipe, the operator proceeds as follows:—

α. A part of the substance to be examined is gradually heated to redness, in a small glass retort, over the spirit lamp. During this process, all phenomena must be observed,—if decrepitation takes place,—if water or any other volatile body be eliminated, and its odor. The water condensed in the cooler parts of the retort should be tested by litmus or Brazil wood paper, to ascertain if it gives an acid or an alkaline reaction.

β. Another portion is heated gently upon charcoal; the odor will indicate volatilized acids, arsenic, selenium, or sulphur. The difference of smell should be noticed, when the sample is heated in the oxidating as well as in the reducing flame, since selenium and sulphur are best detected in the former, and arsenic in the latter. It should be noticed if the charcoal has become coated with a sublimate, and if near or at a distance from the assay; of what color it is, and if it remains the same when cold as when hot. If the substance under examination be an earth, it must be heated strongly for some time, and then removed from the

charcoal to litmus paper, and moistened with a few drops of distilled water ; if an alkaline reaction occurs, the substance contains a carbonate of one of the alkalis ; for these occur in nature,—*Witherite, Strontianite, Calcareous spar. Magnesite, Bitter-spar, et cetera*,—and become caustic with strong heating, and exhibit alkaline properties.

γ. If the mineral contains volatile metals, metallic oxides, sulphur, *et cetera*, it is examined in a tube which is open at both ends. The substance is placed within the tube, at a short distance from one end, which is then heated with the spirit-lamp, and afterwards with the Blowpipe flame. The tube is held in an inclined position. The more perpendicular the position, the stronger the current of air. It is very easy to regulate the rapidity of the draught according to the rate of oxidation that may be required.

In this treatment, many substances are sublimed, which were not when heated in the retort, because the access of air was comparatively small. The oxygen of the air is absorbed, forming volatile acids or metallic oxides ; some escape as gases, and may easily be recognized by their smell,—sulphur, for instance, exhaling sulphurous acid. Others are deposited in the upper and cooler parts of the tube, at greater or less distances from the heated body, according to their degrees of volatility ; in the cases of arsenic and antimony, for example, the former is given off as arsenious acid, and the latter as oxide of antimony.

δ. In examining a substance with regard to its *fusibility*, a part is placed in a small cavity, formed in a piece of charcoal, and heated for a length of time in a strong oxidating flame. The more easily reduced metals and metallic oxides are acted upon in this experiment.

If the substance be an earth or silicate, small pieces of it are broken off with a hammer, and a fragment chosen with a sharp point or side. This is held between the platinum points of a pair of forceps, and a strong Blowpipe flame allowed to play on the sharp extremity for some time. It will at once be seen if the

substance is fusible. If infusible, the sharp point or edge will remain unaltered ; if fusible, it becomes rounded ; and if entirely fusible, it will melt into a round bead. These appearances should be examined through a strong magnifying glass. When a substance is heated in the inner flame, the outer may become colored ; for instance, if the mineral contains potassa, the outer flame will be violet ; if soda be present, the flame appears yellow ; whilst lithia communicates a red tinge. If the three are together, *the yellow flame predominates*.

BERZELIUS treated those minerals which were found to be very infusible, in the following manner :—The substance is levigated in an agate mortar, with water. The menstruum, with the finely divided particles floating through it, is dropped on the charcoal, and heated by the oxidating flame, to evaporate the water. This operation is repeated until the charcoal becomes covered with a coating of the fine particles, which are carefully collected and held in the points of the forceps, and then subjected to a powerful oxidating flame. For the most part, some change takes place, owing to the extreme divisibility of the mineral, showing that the substance is not altogether infusible. In this case, the microscope will be in great requisition, in order to discern the action which may have been produced.

When a mineral occurs in a granulated state, such as sand, one of the grains should be placed on the charcoal ; but as this is so apt to be lost, the better way is to knead the substance with water into a paste, which is then placed on the charcoal, dried by the oxidating flame, and examined as to its fusibility. This process should always be resorted to, when treating substances that are in a minute state of division.

The heat obtained by blowing through the small hand Blowpipe is limited, so that there are some bodies which are never fused before it ; silica and alumina, for instance, in however finely divided a state. Many minerals melt and tumefy, and thereby give a blistered glass, which, on account of the air-bubbles, appear opaque. The intumescence and blistering appear at a certain degree of heat, that is, when all the water is expelled. According to BERZELIUS, the cauliflower appearance which some-

times takes place on heating, proceeds from a change in the combination of the ingredients and their relative positions; but the tumefaction and blistering which occur in igneous fusion, he considers to arise only from the escape of some ingredients in the form of a gas, although it often happens that this ingredient is not discriminated in an analysis of the body. This occurs for the most part with silicate of lime or alkalies in combination with alumina.

The examination of the comparative fusibility of minerals is of essential importance, as many which consist principally of earths, and contain very little of the true metallic oxides, can readily be distinguished by this means. Hence it is that the list given by Ross, in all the editions of his *Manual of Analytical Chemistry*, of a considerable number of minerals arranged according to their different degrees of fusibility, is of great interest.

Of the minerals which occur most frequently, the following are, when heated between the platinum points of a forceps in a strong flame, perfectly infusible:—*Quartz, Corundum, Spinel, Zeylanite*—*Pleonaste*—*Automolite*—*Gahnite*—*Olivine, Cerite, Zircon, Diathene*—*Cyanite*—*Leucite, Talc, Gehlenite, Anthophyllite, Staurolite, Allophane, Kymophane, Gadolinite*. Those that phosphoresce on being heated:—*Rutile Titaneisen*—or *Titaniferous Oxydulated Iron*—*Tantalite, Turquoise*—*Calaito*—*Chondrodite, Topaz*. Very difficultly or only on the edges fusible, are particularly the following:—*Adularite, Tartarine*—*Albite*—*Petalite, Labradorite, Anorthite, Tabular Spar, Meerschaum, Speckstein, Serpentine, Epidote*. Those which tumefy on the first application of heat:—*Dichroite*—some varieties moderately fusible—*Beryl*—*Emerald*—*Euclase, Titanite, Sodalite, Schwertstein*—*Tungstate of Lime*—*Heavy Spar*—*Sulphate of Baryta*—*Celestine, Gypsum*—*Sulphate of Lime*—*Apatite, Fluorspar*. The fusible are—the *Zeolites*, the most of which present intumescence when heat is first applied, *Oligoklas*—*Soda Spodumene*—*Spodumene*, which also tumefies, *Meionite, Elaeolite, Nepheline, Amphibolite*, the greater part of which effervesce during fusion. The *Pyroxenes*, of which those containing large quantities of magnesia are with difficulty fusible, *Vesuvian, Idocrase*, which

tumefies on melting, *Orthite*, which boils while fusing, *Wolfram*, *Boracite*, *Datholite*, *Botryolite*, *Tourmaline*, and *Azinite*, which swell up when melted, *Amblygonite*, *Lazurstein*, *Hauyn*, *Nosin*, *Eudyalite*, and *Pyrosmalite*.

c. The comportment of the substances with fluxes should be examined. The fluxes are BORAX, MICROCOSMIC SALT, and CARBONATE OF SODA.

§ 1.—TREATMENT OF THE SUBSTANCE WITH BORAX.

This can be performed either on a platinum wire or on charcoal. If the substance contains only earths and metallic oxides, it should be first examined on the wire. The following is the method of procedure: the loop of the platinum wire is first heated to redness in the Blowpipe flame, then rapidly dipped into the borax, and the adhering particles fused in the oxidating flame. As it seldom happens that a sufficient quantity of borax adheres the first time, the process should be repeated once or twice, until a bead is formed large enough to fill the loop completely. The bead generally remains so firmly fixed in the loop, that it cannot be removed without force. If the wire is quite clean, the bead will be perfectly colorless, both when hot and cold; if it should happen to be slightly tinged, which is easily perceived by holding it between the eye and the light, it must be removed from the platinum wire. The separation of the glass bead from the wire is easily effected, by heating the bead strongly, and then quickly removing it from the flame, placing it over a porcelain basin, and striking the wire against the rim; it immediately falls into the basin and solidifies. The quicker this operation is performed, and the firmer the wire is held, the more perfectly is the separation of the bead effected.

The borax bead melted on the platinum loop, must, when cold, be moistened on one side with the tongue, which side should then be brought in contact with the substance to be tested, in the state of fine grains, or of very fine powder, and the adhering particles melted, together with the borax, in the oxidating flame. In

general, earthy substances are better examined when a single particle is at first dissolved, because when the substance is employed in the state of powder, the undecomposed part cannot be well distinguished from the separated and insoluble portions which it may contain. With borax this is not so often the case as with microcosmic salt, for with the latter silicic acid is separated.

When the oxidating flame is playing upon the assay, the operator must observe if it dissolve quickly or slowly; whether any gas is generated; if, when entirely dissolved, the glass when held between the eye and the light is colored; and whether the color changes or remains the same on cooling.

There are some bodies which, with borax in certain proportions, give a clear glass when hot or cold, but in the reduction, or more particularly in a quick intermittent flame, become opaque, milk-white, or opalescent, and, in some cases, even colored. This reaction is generally observed in such bodies as form clear glasses while melting, but are enamelled on cooling. Such is the case with the alkaline earths, with yttria, glucina, zirconia, oxide of cerium, tantalic and titanic acids, and with some others, as silica, alumina, sesquioxide of iron, oxide of manganese, *et cetera*. The presence of silica hinders the phenomenon of the glass becoming enamelled on cooling. The same occurs with those silicates which give a transparent glass only when the assay is perfectly saturated,—oversaturated, the mass is, on cooling, opaque. When a bead of borax, containing a body in certain proportions, becomes opaque in a quick intermitting flame, it is said to become *turbidly streaked*.

Where metallic oxides are present, and the glass may be colored, it is necessary to observe that too much of the sample be not taken; otherwise the color will be so deep as to be indistinguishable. When a glass is so dark that the hue cannot be decided upon, it must be pressed while hot between the points of a forceps, or drawn out into long thin threads; the color can then be more readily recognized. Many judge of the color best with the naked eye; others with a magnifying glass: but in the latter case, it must be particularly noticed that the glasses of the magnifier are not tinged, else false determinations will be the result.

After the color which a substance gives with borax before the oxidating flame has been remarked, and all the phenomena carefully observed, the glass must be operated upon in the reducing flame, and in such a manner that no carbon be deposited on its surface. If there appear to be metallic oxides dissolved in the glass, which are not reduced by the borax, as for example, oxides of cerium, manganese, cobalt, iron, uranium, chromium, titanate and tungstic acids, *et cetera*, the operation must be effected on the platinum wire : but if metallic oxides are present, which are readily reduced to the metallic state, as the oxides of zinc, nickel, cadmium, lead, bismuth, copper, silver, antimony, *et cetera*, the reduction must be produced on charcoal ; otherwise, the metal would amalgamate with the platinum.

When a trial is made upon the platinum wire, in the two flames, the change of color in the glass will be readily recognized, either when hot or cold ; but if the assay be heated on charcoal, the glass must be removed from the wire as previously mentioned.

The bead is placed in a small cavity formed in the charcoal, and then exposed to the reducing flame. After continuing the flame for a few minutes, the glass is quickly removed, and pressed between the broad ends of the forceps, in order that the color may be more accurately perceived. Pressing the glass in this manner also possesses this advantage, that the protoxides, if there be any present, are not so liable to become sesquioxides, as when left to cool gradually upon the charcoal.

If the glass contains an easily reducible metallic oxide, it often occurs that the charcoal becomes coated with a sublimate at a certain distance from the assay ; but this appearance takes place only when the oxides of selenium, tellurium, arsenic, antimony, bismuth, cadmium, zinc, tin, and lead are in excess.

When no color is given with the reducing flame on the wire, an examination should be made with borax on charcoal, in the following manner. The hot glass is suddenly shook on the charcoal, and heated with a small quantity of pure metallic tin, and the mixture then strongly smelted in the reducing flame. The tin, having a great affinity for oxygen, abstracts part from

the metallic oxide contained in the assay, and dissolves without tinging the glass, while the metallic oxide, *which is converted into a protoxide, gives a characteristic hue to the glass on cooling.* This method is generally resorted to when examining for traces of copper or iron.

When the behavior of a substance containing a sulphide or arsenide is to be ascertained by heating with borax, it is best to sublime, first, most of the sulphur or arsenic, in an open tube, and then to heat on charcoal, with the necessary proportion of borax, and tin if requisite, in both flames, and observe all the striking characteristics. When sublimation-tubes are employed, they should be free from *lead*, for arsenic, if heated in a tube containing this metal, decomposes the glass.

§ 2.—TREATMENT OF THE SUBSTANCE WITH MICROCOSMIC SALT.

As with Borax, it can be treated partly on the platinum wire, and partly on charcoal. If the substance contains earthy ingredients, or only difficultly reducible metallic oxides, the operation is to be conducted on platinum wire, in exactly the same manner as with borax; but if it likewise comprises sulphides or arsenides, it must, as with borax, be treated on charcoal. As microcosmic salt gives a very fluid bead when heated, a few additional bends should be made on the platinum wire, when this salt is used, to prevent the melted drop from falling; on charcoal, however, enough of the salt can be taken at once, as there are no obstacles in the way. This flux is better for many metallic oxides than borax, as the colors are often better ascertained when a sufficient quantity of the substance is dissolved. It also separates the acids;—the volatile are driven off, and the fixed remain, and divide the bases either with the phosphoric acid, or remain uncombined, and float about in the molten mass. With the silicates, this is particularly the case: the silicic acid is separated, and flows through the glass in a gelatinous state. Silicates, when containing bases that, by themselves, produce in vitreous fluxes an opaque glass on cooling, give, in combination with borax, a clear glass; but an opalescent, with microcosmic salt.

§ 3.—TREATMENT OF THE SUBSTANCE WITH CARBONATE
OF SODA.

When assaying a substance with soda, it is necessary to learn if it consists of earthy ingredients only, or of acids, free or in combination; whether it is soluble; whether it contains metallic oxides which give a reduced metal; or if sulphides or arsenides are present, *et cetera*. The numerous examinations will be fully treated of, together with the pyrognostic effects produced upon metallic oxides, when heated in the Blowpipe-flame with Carbonate of Soda.

a. Fusibility of a Substance with Carbonate of Soda.

A great number of bodies possess the property of uniting with soda, and forming combinations partly fusible and partly infusible: to the former, but few belong, such as silica, and the metallic oxides of titanic, tungstic, and molybdic acids, *et cetera*; they are, however, more or less absorbed by the charcoal, if not in combination with silica.

In testing the fusibility of a substance with Carbonate of Soda, the following is the mode of procedure:—

The substance, when in powder, is kneaded into a paste with soda, a moistened spatula being used for the purpose; but when in a granular state, one of the grains is taken, and covered with the soda paste; in both cases a portion is placed on charcoal, and heated till all water is expelled; the mass is then strongly ignited in the oxidating flame. The carbonate of soda will first be absorbed by the charcoal; but when a sufficient quantity of the assay dissolves, it exudes, effervescence takes place, and a bead is formed. The treated substance, if insoluble in carbonate of soda, will, if decomposed, tumefy gradually, and change its appearance, without forming a bead. The operator should add fresh supplies of the flux, and renew the blast until he arrives at a satisfactory conclusion. When, to a substance, soluble in this flux, too little of it has been added, a part will remain opaque, surrounded by a clear glass; if too much carbonate of soda has been supplied, the glass will be quite opaque; it is therefore always advisable that

the carbonate of soda be supplied in small doses at first, and more added as the different changes ensue. If the glass assumes a yellow, red, or brown color, sulphur or sulphuric acid has been present; the smallest trace of these substances gives a decided reaction. When the substance has been examined on charcoal, it should be treated on the platinum wire in the oxidating flame; if it expands and gives a bluish-green glass in cooling, manganate of soda has been formed, and, therefore, some oxide of manganese has been present. Should the substance contain silica and oxide of cobalt, silicate of soda is formed, and the glass assumes the characteristic cobalt-blue color. When the previous reaction has been decided upon, and an operator wishes to ascertain the presence of an alkali or earth, the substance must be treated upon platinum foil, with carbonate of soda. If the glass during the fusion is clear and limpid, only an alkali is contained therein, but if something is deposited, an earth is present. The alkali will also tinge the flame, which must be remarked.

b. Reduction of Metallic Oxides by means of Carbonate of Soda.

In this process, metallic oxides, existing in only very minute quantities in minerals, *et cetera*, may be more readily found than by the moist way.

There are some metals which, without the addition of carbonate of soda, are reduced on charcoal in the reducing flame, and thus determined; but if they are mixed or in combination with other substances irreducible, it is not only difficult, but, in many cases, altogether impossible, to accomplish the reduction sufficiently, in order to arrive at satisfactory results. For instance, when a quantity of native or artificial oxide of tin is treated in the reducing flame upon charcoal, it is possible, with great exertion and fatigue, to obtain a metallic globule; but when carbonate of soda is used as an auxiliary, the reduction takes place readily.

When a metallic oxide is in combination with a non-reducible body, which prevents its giving a characteristic reaction with carbonate of soda, it must be treated as the annexed process directs.

Knead the powdered sample, with moist carbonate of soda, into

a paste, then place the mass on charcoal, and direct upon it a strong reducing flame. The flux will at first be absorbed; but more must be added, and the heat continued as long as any portion of the assay remains on the surface of the charcoal. The first portion of carbonate serves for collecting the metallic contents, while the latter is for completing the reduction of the remaining unreduced metallic oxide. A few drops of water are now added to extinguish the ignited parts, and then the whole of the charcoal upon which the assay rested, is cut out with a knife, and put into an agate mortar, care being taken to lose nothing in the transfer. The mass is mixed with water, and ground to a fine powder. If there be any metal, from its greater specific gravity, it falls immediately to the bottom. Repeated levigations and decantations are then resorted to, till the whole of the pulverized charcoal is removed. When the charcoal is entirely washed away, the metal, if any had been in the assay, will be found at the bottom of the mortar. If it is an infusible or a brittle metal, it appears in the form of a metallic powder; if a malleable one, it produces flat shining plates.

By this method, half a per cent. of *Tin*, and even a much smaller quantity of *Copper*, may be very easily detected. If, however, a number of metallic bodies are contained in the same substance, they will be reduced, together, to a metallic alloy. Some, notwithstanding, are obtained singly, as copper and iron, which give reguli distinct from other metals.

The metals which can be reduced by this means, besides the noble ones, are,—Molybdenum, Tungsten, Antimony, Tellurium, Lead, Zinc, Tin, Copper, Bismuth, Nickel, Cobalt, and Iron. The last three are magnetic, and can therefore be at once distinguished from the others. However, there are a few of these which are partly or altogether volatile, and coat the charcoal with sublimated oxides; namely, Antimony, Tellurium, Bismuth, Lead, and Zinc. Some other metals, as Arsenic, Cadmium, and Mercury, are also reducible, but, being extremely volatile, must be heated in glass tubes, in order to obtain their sublimate.

If, in these experiments, a metallic button, containing a number of metals, is obtained, the alloy must be examined on char-

coal, with borax or microcosmic salt, as will be hereafter adverted to when treating of different qualitative metallic assaying.

When the substance under examination is a sulphide or arsenide, or contains either sulphur or arsenic, these bodies must in the first place be driven off, and the metal oxidized, before the reduction test can be made. The process is conducted as follows: the sample is ground to an impalpable powder, and pressed into a shallow cavity in the charcoal with the spatula, then acted upon by the flame. The oxidating flame is to play upon the assay first, by which means part of the sulphur is given off as sulphurous acid, and the metal oxidized, which combines with the sulphuric and arsenic acids, also formed in the process. When no more sulphurous acid is evolved, which is readily recognised by its characteristic odor, the reducing flame must be brought into action, whereby all sulphuric and arsenic acid is reduced, and the greater part of the metallic arsenic is expelled. When the alliaceous smell is no longer discernible, the oxidating flame is again resorted to, until decomposition is so far accomplished as to satisfy the operator. The assay is now turned over on the charcoal, and the other side treated in the same manner. The mass is then removed, again powdered, and once more treated on the charcoal with the oxidating and reducing flames. The sulphur is by this means entirely got rid of, but arsenic frequently remains as arsenic acid, in combination with such oxides as those of cobalt or nickel, and can only be removed with great difficulty. When the substance contains a large proportion of arsenic, which might be dangerous if it were allowed to escape in the room, previously to roasting, it should be treated in a tube, by which means the greater part of this metal sublimes, and condenses in the upper part.

If the substance contains neither cobalt nor nickel, but another metal with arsenic, the metal can be obtained by the reduction of the roasted sample with soda, perfectly free from the arsenic; but if cobalt or nickel be present, the reduced metal will always be contaminated with arsenic, and must therefore be further treated *with borax* on charcoal. In this reduction experiment, an operator must allow an uninterrupted strong flame to play

upon the assay. Great care must be exercised in separating the charcoal which has become impregnated with reduced metal, and also in the levigation and decantation ; and, lastly, the metal obtained in grains, plates, or fine powder, must not only be inspected with the naked eye, but also through a magnifying glass.

When the substance is an earth, containing no coloring metallic oxides, it is to be treated with a solution of nitrate of cobalt. A piece is selected, of sufficient porosity to allow the solution of cobalt to permeate, and a few drops added. It is then subjected, in the points of the forceps, to a moderate oxidating flame ; during this process, the color will assume a characteristic aspect—if blue, pure alumina is indicated ; if rose-red, magnesia. If the latter color is discernible, the assay must be fused, because, when magnesia is present, the rose-red tint is better brought out in a strong flame. Minerals containing lime, or an alkali, give a blue tinge, when *strongly* heated with cobalt ; but alumina gives it by a moderate heat, and can, therefore, be readily distinguished.

Those crystalline rocks which will not admit of the solution permeating, must be reduced to a fine powder in an agate mortar ; then kneaded into a paste with water, and spread on the charcoal. The solution of nitrate of cobalt is now added, and the mass subjected to an incipient red heat. If decomposition does not take place, the color imparted,—blue, red, or black, proceeds from the cobalt solution only, and not from any of the ingredients. If the mass separates from the charcoal, after the moderate red heat, as a thin pellicle, it must be submitted to an intense oxidating flame, in the points of the forceps. The color now imparted must be observed on cooling, by daylight ; because, when seen at candle-light, a blue often appears dirty violet, or even red, by transmitted light.

When a metallic oxide is present in a mineral, which may give a colorless result, it will not interfere with the alumina or magnesia reaction, unless a large quantity is present, and then the mass will generally become grey or black. In treating some minerals *per se* on charcoal, or in the reduction test with carbonate of soda, the metal often sublimes, becomes oxidized immediately, and is deposited upon the charcoal. Many such oxides

are easily expelled by an oxidating flame, others with greater difficulty, and some not at all ; so that it is often almost impossible to separate the pellicle of oxide from the charcoal. If such a film rest upon the support after the experiment, a few drops of a solution of nitrate of cobalt is to be added to it, and the assay submitted to a pretty strong oxidating flame. If zinc be present, a distinct bright green color will be imparted to the substance, which should be viewed through the microscope.

The quantity of the cobalt solution to be employed depends greatly upon its strength : experience, however, very soon teaches the proper quantities to be taken, in order to obtain a decided reaction.

Minerals, *et cetera*, examined according to the foregoing rules, in flasks, open tubes, and with fluxes, in the different flames produced by the Blowpipe, give numerous phenomena from which results must be gathered ; and attention must be given to the slightest occurrences that ensue, for such often lead to the discovery of substances, *never supposed to exist in the assay under examination*.

The results, however, obtained in the examination of an unknown body before the Blowpipe, can be fully appreciated only by those who are acquainted with chemistry, and, therefore, no rules can be laid down sufficient to detail all the minutiae, as they can be arrived at by industry and practice only.

¶ B *General Rules for Qualitative Blowpipe Analysis, by means of which, partly with the assistance of the moist way, the single ingredients of compound bodies may be detected.*

If by means of the Blowpipe, alone, or with glass fluxes, the constituents of a compound body are not satisfactorily determined, the moist way must be resorted to, in order to ascertain, as nearly as possible, the collective ingredients. By such an association an operator can, with accuracy and facility, examine the generality of bodies composed of different earths and metallic oxides, native silicates, slags, *et cetera*.

For the manipulation, 100 to 150 milligrammes of a substance are quite sufficient to take, in order to detect all the ingredients; and should the operator be an expert Blowpipe analyst, he will find that less will serve—only a small portion of a liquid reagent is necessary. When its pyrognostic qualities have been remarked, or the class to which it belongs has been determined, and whether it is an alkali,—earth, metallic salt, silicate, aluminate, compound metallic oxide, metallic oxide with an earth, metallic sulphide,—or a combination of different metals, including arsenic and selenium, the single ingredients may readily be recognized. The procedure will be particularly adverted to in the qualitative analysis of alkalies, earths, metallic and non-metallic bodies, and such substances as are met with in nature. To prevent a repetition of many manipulations, we shall previously describe the methods for melting and dissolving, *et cetera*, and afterwards give them separately.

§ 1. SMELTING OF THE SUBSTANCE WITH BORAX AND CARBONATE OF SODA.

A compound in which the earths cannot be detected by a single reaction, must be triturated in an agate mortar to an impalpable powder, and from 75 to 100 milligrammes employed for the analysis. If it contains sulphides or arsenides it must be roasted twice with charcoal, in a manner similar to the quantitative assaying of copper, to expel all or the greater portion of the sulphur or arsenic, and to oxidize the metals which are generally combined with traces of formed sulphuric or arsenic acids. The powder is now triturated in a mortar with carbonate of soda and borax, the proportions of these reagents being rectified according to the degree of solubility of the assay; the mixture is then pressed in a cornet of soda paper, similar to a quantitative copper assay, and operated upon in a cylindrical cavity made in a piece of charcoal, by an intense Blowpipe-flame. In general, the melting is more readily effected in the oxidating than in the reducing flame. If sulphuric acid or reducible metallic oxides are not present, the oxidating flame is always preferable, but if otherwise,

the reducing flame,—in which the metallic oxides sublime, and the sulphuric acid is reduced to sulphur, which combines with the sodium, *&c cetera*.

When the metallic oxides are in such small quantities as to be difficultly reducible to separate beads, 200 or 300 milligrammes of lead are added, or else a globule of silver, weighing 80 to 100 milligrammes, and the process is carried on like a quantitative silver examination. By this method, the earthy materials, and difficultly reducible oxides, undergo the igneous fusion, and form a light liquid pearl. The sulphuric acid, acids of arsenic, and easily tested oxides are reduced, if present; the sulphur combines with the sodium and some of the other metals, the metallic arsenic partly sublimates and is partly absorbed by the alloy, and the non-sublimable metals unite, particularly when lead or silver has been added, forming a globule. The metallic globule attaches itself to the glass, especially if lead is predominant, a part of which volatilizes. The metallic oxides dissolved in the glass are principally in the state of protoxides. An intense oxidation or reduction must be employed for the preceding analysis, because otherwise fluidity, or the reduction of the different ingredients, would not ensue. The smelted assay must be obtained limpid, and be as free as possible from bubbles and metallic particles. When much magnesia is present, it is difficult to obtain a translucent glass, therefore more borax is to be added. If, after blowing for some time, metallic laminæ are still perceived, it indicates reduction of the oxides, or solution of the non-reducible parts; therefore the heat must be continued.

When an analysis is completed, the assay must immediately be removed from the charcoal, by tapping it upon an anvil or on paper, to an agate mortar, and pulverized. This precaution is necessary, because the ignited mass readily absorbs moisture, becomes unctuous, and is therefore powdered with difficulty.

When the assay has been fused in the reduction flame, whereby metallic globules are obtained—compound alloys, containing the lead or silver added with other metals,—these must be separated from the molten mass, by allowing it to flow gently from side to side upon the charcoal, while covered by the reducing flame.

The whole is then removed with a spatula, and the carbonaceous particles either burnt or scraped off with a knife. The glass is then separated upon the anvil from the attached metallic globule. If the necessary reduction heat is not attended to, part of the metallic oxide may remain, or a trace be oxidized through the medium of the lead, and dissolved in the glass, which may have such an influence on further manipulation as to give entirely false results.

The metallic oxides which are easily reducible with carbonate of soda and borax in the reducing flame, and which can readily be separated from certain earths and other irreducible metals, are as follows :—Arsenious, Arsenic, Antimonious, and Antimonic Acids ; Oxides of Antimony, Silver, Mercury, Copper, Bismuth, Tin, Lead, Cadmium, Zinc, and Nickel. Selenium, Tellurium, Osmium, Gold, Platinum, Iridium, Rhodium, and Palladium are never met with in nature, but in the metallic state ; they are, therefore, readily separated from certain earths and metallic oxides by cupellation with lead or silver. Those metals which are volatile, evolve, either partly or entirely, a vapor by such fusion, which condenses in the charcoal. The metallic oxides which are not reduced when melted with carbonate of soda and borax, are—Sesquioxide of Chromium, Molybdic, Tungstic, Tantalalic, and Titanic Acids ; Oxides of Uranium, Cobalt, Iron, Manganese, and Cerium. These can, however, be readily separated from the earths, and recognized by their pyrognostic effects, as will be adverted to under the different heads.

§ 2. METHOD OF TREATING WITH HYDROCHLORIC ACID THE SUBSTANCE MELTED WITH CARBONATE OF SODA AND BORAX.

The powdered glass is removed to a porcelain basin, and then affused with an excess of hydrochloric acid. The menstruum is now to be stirred with a glass rod, and the whole heated upon the triangle, over the flame of the lamp. By this treatment soluble and insoluble parts are separated, the sulphide of sodium is decomposed, the sulphur combines with the hydrogen of the acid, and is eliminated as sulphide of hydrogen—hydrosulphuric

acid,—and the other substances, except the silicic acid, form metallic chlorides. But as silicious combinations only are generally separated by fusion with carbonate of soda and borax, consequently, in these cases the operator very seldom encounters Molybdic, Tungstic, Tantallic, and Titanic acids.

So soon as the solution is completed, the whole menstruum is to be evaporated slowly to dryness, under a chimney, in order to prevent the metallic chlorides, principally composed of chloride of sodium, from being lost by decrepitation.

The evaporation serves two purposes,—to expel the excess of acid, and to separate the silicic acid—silica—as gelatinous hydrate. When evaporated to a consistency, distilled water is added, and the whole mixture again heated ; whereby the metallic chlorides are dissolved and separated from the insoluble residue. If the residue contains silica, it can readily be recognized, by filtering, washing, and treating what remains with microcosmic salt, or carbonate of soda, before the Blowpipe.

If the substance contains sesquioxide of iron, it becomes reduced, when smelted, to the state of protoxide ; and is not, on the addition of hydrochloric acid, again converted into the sesquioxide. As it is, however, absolutely necessary, in order to detect accurately the different constituents, that the iron should be in the state of sesquioxide, a few drops of nitric acid are to be added to the filtrate, from the silicic acid mixed with the first water used in the edulcoration, and heated, in order to accomplish the purpose. The different constituents intended for examination are then separately investigated, according to the methods given under their respective heads.

§ 3. SMELTING OF THE SUBSTANCE WITH NITRATE OR BISULPHATE OF POTASSA.

When only one of the constituents of the substance under examination is sought, it is sometimes necessary to fuse it with saltpetre, in order to convert that constituent into a higher oxide ; or, in case it be an acid, to combine it with the potassa of the saltpetre, from which it can be easily separated and recognised.

In the same manner a substance is often fused with bisulphate of potassa, and the melted mass dissolved in water, in order either to free it immediately from certain constituents, or to convert the whole into sulphates, and thus be able, after the solution in water, to effect the separation of its different components.

Fusion with saltpetre may sometimes be performed in the loop of the platinum wire, but it would be better to employ a platinum spoon. The surface of platinum is oxidized slightly by saltpetre ; but this is of no moment, as it produces no disadvantageous effects on the experiment, and the destruction of the spoon need not be feared. Pulverulent and pulverizable substances should be mixed in a finely triturated state, in a mortar, with the necessary quantity of saltpetre. Metallic alloys, which do not admit of being pulverized, must be reduced to a finely-divided state either by the hammer or file. The quantity of saltpetre to be used can be ascertained from the facility with which the substance undergoes oxidation ; from three to four times the bulk of the substance may generally be employed, provided it does not possess a great specific gravity. When one constituent only is sought, the fusion may take place on the platinum wire, but the mixture must in such a case be moistened with a little water, and in a soft or doughy state be placed on the loop. The oxidating flame alone is used in the fusion, and when the mass has ceased to vesiculate, another portion of the moistened mixture is to be added to the fused mass, and both melted together : this is to be continued until the molten mass, owing to its size, can no longer remain attached to the wire. During the fusion the wire must be held obliquely, and in such a manner that the loop is directed downwards ; as, otherwise, the saltpetre would be apt to flow along the wire.

If it is conjectured that the substance contains only an extremely small quantity of the body sought, or if it be intended to oxidize other constituents, in order to be able, in this state, to subject them to further treatment,—or if a metallic alloy, or an unpulverizable substance be under examination,—a larger quantity must be taken for the experiment, and the fusion performed in a platinum spoon. The whole mixture should not, however, be put into the spoon at one time, but only small portions ; be-

cause, during the fusion, gas and vapor are given off, which would easily cause the melting mass to swell and flow over the margin. The spoon should be first treated externally on the bottom with the oxidating flame ; the flame should then be directed into the substance, and the whole fused until it becomes perfectly fluid ; the remainder should then be added to it in small portions, and the mass perfectly liquefied after each addition. During the fusion, the position of the spoon before the oxidating flame should be so changed, that every part of the mass to be fused be subjected to its action, and the spoon itself raised to a red heat.

Such metallic alloys only can be treated with saltpetre in a platinum spoon as are readily oxidized, and do not combine with the platinum at a temperature capable of being produced by the Blowpipe. The fusion of a metallic alloy with saltpetre is thus confined to the detection of traces of arsenic, in such metals as can with difficulty be separated from it, and those which are exceedingly difficult of fusion, as, for example, nickel.

The fusion of a substance with bisulphate of potassa may also be performed in a large platinum spoon, either in the same manner as with saltpetre, or still better in the flame of the spirit lamp ; the substance must, however, be perfectly dry, and in a finely divided state. When the fusion is made with the Blowpipe, some of the sulphates formed by such a manipulation may be again deprived of their acid by the strong heat. If, however, the fusion is performed in the flame of the spirit lamp, the spoon in the commencement being held only over the top of the flame, until the greater part of the gases escape, and then sunk deeper in the combustible, so that the heat may act equally from all sides on the bottom of the spoon, the melting mass will be raised to an incipient red heat only, and the salts so formed will not be decomposed. In some cases, where a large quantity of the salt must be employed, the spoon becomes filled before the whole of the mixture has been added. In this case the fluid mass should be poured out on the anvil, and the remainder of the mixture then melted. It is also sometimes advisable to pour out the melted mixture, particularly in the examination for titanous acid, where the water necessary for its solution cannot be raised to the boiling point. The quantity of bisulphate of potassa which should be

employed, depends upon the different constituents of the substance ; thus, for example, for protoxide of iron, three to six times ; for lime, four or five times ; for magnesia, six times ; and for alumina, seven to eight times the weight of bisulphate is required to convert them into sulphates. The operator always proceeds more cautiously by employing a slight excess of the acid salt, as the oxides of iron, and alumina, by continued high temperature, lose with great facility a part of their combined sulphuric acid.

§ 4. SOLUTION IN WATER OF THE SUBSTANCE FUSED WITH SALT-
PETRE OR BISULPHATE OF POTASSA.

The solid mass obtained by fusing a substance with saltpetre or bisulphate of potassa, whether on the platinum wire or in the platinum spoon, cannot, unless it has been poured out when in a state of fusion, be well pulverized, and in this state dissolved in water, because in the removal of such a mass the platinum is liable to be tarnished ; it is, therefore, necessary to place the wire or the spoon with the fused mass in a porcelain basin or cup, proportionably to the volume of the mass, then pour over it the quantity of water necessary for its solution, and place the vessel on the triangle over the flame of the lamp. As the water becomes warm, the mass generally separates from the platinum, and can then be crushed with the pestle of the agate mortar. In most cases the water can be raised to the boiling point, and the mass thus easily dissolved ; but if a titaniferous mineral be melted with bisulphate of potassa in order to render the titanic acid soluble, the water must not be raised to the boiling point, as otherwise the titanic would not be dissolved, and even that part which was, at a low temperature, would be again precipitated. The manner of treating the solution, and the residue which could not be converted into sulphates, will be more fully entered upon hereafter, when we come to speak of the qualitative examination of earths and metals.

§ 5. PRECIPITATION—BY CRYSTALLIZED OR PULVERULENT RE-
AGENTS—OF SINGLE CONSTITUENTS DISSOLVED IN FLUIDS.

If it be necessary to decompose a clear solution with a dry salt, the acid or base of which is capable of combining with one of the

constituents of the dissolved salt, and as a compound insoluble in this fluid is precipitated, the fluid must in most cases, after the addition of the salt, be slightly heated, in order that it might easily dissolve, and thus serve as a reagent. In some cases it is also better to warm the liquid before the addition of the salt, as in this case the precipitate is instantly produced. Thus, for example, baryta and strontia can be precipitated by pulverized bisulphate of potassa; lime by crystallized oxalate of ammonia; magnesia by microcosmic salt in crystalline grains; phosphoric acid by a crystal of nitrate of silver; chromic acid by acetate of lead, *et cetera*. The quantity of the reagent used each time entirely depends upon the quantity of the substance to be precipitated.

§ 6. DECANTATION, FILTRATION, AND EDULCORATION.

In cases where only one substance is to be sought, in the clear liquid which covers a residue or precipitate, it is only necessary to pour off carefully the greater part of the liquid. But if the liquid, or the residue, or precipitate is to be examined for a number of constituents, the whole should be filtered, and the residue on the filter edulcorated with water.

A very thin, porous, or unsized paper should be employed, which should be cut into filters, of an inch or an inch and a half radius. For exceedingly small precipitates, where it may be necessary to remove a portion of the paper with the precipitate, filters should be made of Swedish paper, which is prepared with distilled water, or from paper freed from its earthy constituents by means of hydrochloric acid, next well washed with distilled water, in order to remove the adhering acid and salts, and then dried.

The operator should proceed in the following manner with the filtration and edulcoration. The filter, placed in the funnel, should be saturated with distilled water, in order that as little as possible of the dissolved substance might remain behind in it, the liquid and precipitate, or residue, should then be poured by degrees on it; the glass which contained the mass washed with a little water thrown on the filter, and the whole liquid allowed to percolate. If the filtrate contains a number of substances, which it is intended to examine, the filter should be filled with water, which

dissolves the greater part of the soluble matter still adhering to the filter and its contents ; this water, on passing through, should be added to the first liquid. No more of the water used in the edulcoration should be added to the filtrate, otherwise it becomes too dilute, and the constituents are very difficult to be separated from one another. The funnel, with the filter, should then be placed upon another vessel, larger,—as for example, a flask with a narrow neck,—and the residue well edulcorated with more water. The washing-bottle described at page 33 can be used in edulcoration, in order the better to bring a small precipitate to the bottom of the filter. It seldom happens that boiling water is necessary to wash a precipitate ; cases, however, occur where boiling water may be used with advantage, particularly with argillaceous precipitates, which are to be further treated. In this case, the operation may be performed in the following manner : distilled water should be poured into a porcelain vessel, and boiled over the spirit-lamp ; a portion should then be sucked up with the glass pipette, described at page 32, and ejected on the precipitate ; when this portion has filtered through, the operation is to be repeated until the affusion is complete.

§ 7. DESICCATION OF THE FILTERED RESIDUES AND PRECIPITATES.

Sometimes it is not necessary to dry the residue or precipitate, as it may be subjected, while still moist, to further investigation. Cases, however, occur where it must be perfectly dried, and in this state further treated with fluxes on charcoal, or in the platinum spoon.

If the substance to be dried is in sufficient quantity, the filter should be opened out on two folds of bibulous paper, the mass spread out with a spatula, and introduced into a perfectly smooth porcelain basin, and exsiccated over the flame of the lamp. But if the residue is small, the unfolded filter should be held to the light, and the part of the paper on which none of the precipitate is seen cut off with a scissors, and the remainder, containing the filtered mass, dried in a small porcelain basin. When the paper is dry, it is to be folded double, and inflamed at one end, while firmly held at the other with the forceps, and thus allowed to burn over a clean dry porcelain basin ; into this the residue

gradually falls, mixed with a little carbon, which is easily burned off in the platinum spoon ; but it is unnecessary when the dry mass is to be further treated with fluxes, as it is by this means destroyed.

It would be superfluous to mention here every trifling appliance which so often contributes to the success of a fusion, a solution, *et cetera*, as every person brings to his aid peculiar advantages and manipulation.

REACTIONS BEFORE THE BLOWPIPE OF DIFFERENT OXIDES, ET CETERA.

WE now enter upon a tabulated system, showing the reactions of alkalis, earths, and metallic oxides, alone, and with reagents in the oxidation and reduction flame of the Blowpipe.

When a substance is heated alone on charcoal, the operator, if expert, will readily be able to discern whether he has an alkali, earth, or metallic oxide for his assay. The alkalis and earths will either color the flame yellow, yellowish-green, violet, red, or crimson, or will give some characteristic light or appearance, as fully explained under each head. The metallic oxides will afford globules or grains of metal, brittle or malleable ; *exempli gratia*, lead, silver, bismuth, antimony ; or will indicate copper by yielding a red regulus ; or they will emit a peculiar odor, *vide* arsenic—or tinge the flame,—copper, antimony, *et cetera*.

Another remarkable feature of these compounds is, that they impart to beads of borax and microcosmic salt, most beautiful colors, cobalt giving a deep azure, manganese an amethyst, chromium an emerald-green.

Many substances give most excellent reactions with carbonate of soda, either on charcoal or platinum, or in a test-tube, as cadmium, mercury, manganese, while others yield admirable results with a solution of nitrate of cobalt on charcoal, alumina, and zinc especially.

Heating in a test-tube ought never to be dispensed with by the blowpipist, and this should precede his dry assay, as much trouble may thus be saved. If he heats his substance with lime in a test-tube, ammonia may be evolved, which will be known by its pungent odor ; or if, *per se*, he will ascertain whether a sublimate

is produced, or any peculiar smell developed; if decrepitation takes place; whether the volatile products possess an acid, alkaline, or neutral reaction; if organic matter be present by the assay blackening; and the degree of fusibility.

Examination in the Borax Bead.

As this kind of examination serves particularly for the recognition of metallic oxides, it is promoted chiefly by the conversion of metals, sometimes present in the substance to be tested, into oxides, by a process of roasting. Metals which are combined with sulphur, arsenic, *et cetera*, dissolve not only with difficulty in borax, but exhibit also therewith a behavior differing more or less from that of their oxides.

Before mixing the assay with this salt, it is requisite that the powdered substance be roasted on charcoal or in a glass tube. When the most complete oxidation, as well as the volatilization of sulphur, arsenic, *et cetera*, is to be effected, it must be frequently repeated. Before every fresh roasting, the substance should be rubbed in an agate mortar. Metallic sulphides and arsenides are with advantage subjected alternately to an oxidizing and to a reducing roasting process. The latter is accomplished by mixing the powdered substance with charcoal or graphite in an impalpable state, and then roasting the mixture in a glass tube, or on charcoal. Precisely the same methods are available in the treatment with microcosmic salt, and before submitting the substance to a reaction, it is necessary that the above procedure should be adopted.

Examination in the Microcosmic Salt Bead.

By this examination, as well as by the previous one, the exact observation of the color which the bead shows in the hot condition, while cooling, and when cold, and also in the oxidation and reduction flames is obtained. Beads in which certain oxidized bodies have been dissolved, possess the property of becoming turbid or opaque by sudden changes from a stronger to a weaker blast with the flame of the Blowpipe. This description of blowing is termed *fluttering*. The same result can also be obtained by a slow and gentle heating of the previously cooled bead; the turbidity at last will also arise from increased additions of the substance under treatment.

¶ *Comportment of Alkalies, Earths, and Metallic*

I. ALKALIES

ALKALIES.	Upon Platinum Wire.	
1. Potassa. K O.	When covered with the point of the blue flame, the outer flame is colored violet. A small addition of Soda prevents this reaction.	
2. Soda. Na O.	The flame is colored yellow, although Potassa is present.	
3. Lithia. L O.	If no Soda be present the flame is reddened.	
4. Ammonia. N H ³ .	0	
EARTHS.	Upon Charcoal, and with Borax, on the Platinum Wire.	
5. Baryta. Ba O.	Caustic ; infusible. The Hydrate and Carbonate melt, and are absorbed by the Charcoal.	The Carbonate decrepitates, and forms a bead, which is transparent when cold.
6. Strontia. Sr O.	Hydrate, fusible : the Carbonate, only partly so. The moist soluble salts reddened the reducing flame.	Ditto. With moist soluble salts, the flame is intensely crimson.
7. Lime. Ca O.	Caustic ; not changed. The Carbonate becomes caustic, and emits an intense white light.	Readily dissolved. The Carbonate de-fagrates, and forms, when cold, a transparent crystalline glass.
8. Magnesia. Mg O.	The Carbonate is decomposed, and the residue emits a strong light, and has an alkaline reaction.	Like Lime ; but the bead formed is not so crystalline.
9. Alumina. Al ³ O ³ .	Not changed.	Fuses to a colorless glass, which remains so when cold.
10. Glucina. Gl ³ O ³ .	Ditto.	Fuses to a colorless glass, which becomes slightly opaque by an intermittent flame.
11. Yttria. Y O.		
12. Terbia. T O ³ .		Behaves like Glucina.
13. Erbia. E O ³ .	Assumes a lighter color in the reduction flame.	No striking change.
14. Zirconia. Zr ³ O ³ .	Infusible, but produces a very glaring white light.	Fuses to a colorless glass, which becomes slightly opaque when cold.
15. Thoria. Th O.	Not changed.	With a minute quantity, forms a clear glass.
16. Silicic Acid. Si O ² .	Ditto.	Gives a transparent and colorless bead, which is soluble.

Oxides, alone, and with Reagents, in the Blowpipe Flame.

AND EARTHS.

Upon Platinum Foil.		Remarks.
<p>The only way that Potassa, if contained in a mixture of Potassa and Soda, can be detected by the Blowpipe, is to fuse a clear bead of Borax with a small quantity of Oxalate of Nickel, upon the Platinum wire, and to add the Mixture. If Potassa be present, the brown color of the Nickel bead is changed to blue: a salt of Soda effects no such change.</p>		<p>When in solution, they tinge reddened Litmus Paper blue.</p>
<p>Fuses, with the formation of a dark yellow coating on the surface.</p>		
0		<p>Recognized by its pungent odor. It affects Litmus Paper in the same manner as the preceding alkalis.</p>
With Microcosmic Salt, upon Platinum Wire.	With Carbonate of Soda, on Charcoal.	With a Solution of Cobalt, in the Oxidating Flame.
As with Borax.	Dissolves, and is absorbed by the Charcoal.	Fuses to a yellowish red globule, which is colorless when cold. Becomes greyish by exposure.
Ditto.	Caustic: insoluble. The Carbonate dissolves, and is absorbed by the Charcoal.	Scintillates and partakes of a black or dark grey color.
A colorless bead, which, if saturated, becomes opaque when cold.	Not dissolved. The reagent is absorbed by the Charcoal.	Perfectly infusible, becoming grey.
Ditto.	No action.	Assumes, on cooling, a beautiful flesh-red color.
Fuses to a transparent glass, which becomes slightly opaque, if an excess is present.	Intumescent, with the formation of an insoluble compound. The excess of Carbonate of Soda is absorbed.	Gives a fine transparent blue glass upon cooling.
As with Borax.	No action.	Infusible, but becomes of a bluish grey color.
Like Glucina.	Insoluble.	0
As with Borax.	Ditto.	0
Fuses very slowly; and is scarcely transparent when cold.	No action.	Partakes of a dirty violet color.
As with Borax.	Ditto.	0
Ditto.	Ditto.	With a very small quantity of the Cobalt Solution, a slight blue color is produced, which changes to a dark grey, when an excess is present.

TABLE II.—COMPORTMENT OF

METALLIC OXIDES.	Per Se, on Charcoal.		With Borax on a Platinum Wire.	
	In the Oxidating flame.	In the Reducing flame.	In the Oxidating flame.	In the Reducing flame.
1. Sesquioxide of Cerium. $Ce^3 O^2$.	The Protoxide is converted into the higher oxide. In the Reducing flame, this reaction remains unchanged.		Soluble, forming a red or dark yellow glass. When cool, the glass is only yellow.	The glass containing oxide becomes pale, so that a yellow oxide becomes colorless. When a large quantity of the oxide is present, the glass is on cooling of an enamel white crystalline texture.
2. Oxide of Lanthanium. $La O$.	Unchanged.		Gives an opaline glass.	
3. Oxide of Didymium. $Di O$.	Unchanged.	Becomes grey, losing its brown color.	Amethyst colored glass.	No change.
4. Sesquioxide of Manganese. $Mn^2 O^3$.	Infusible. On being slightly heated, it becomes of a brown color. The Per-, Sesqui-, and Protoxides, produce similar effects.		Intensely colored :—violet—amethyst—while hot; violet, with a tinge of red, when cold. If a large quantity is present, the globule will be black.	The pink bead becomes colorless. Should the color be very intense, the reduction will succeed better upon charcoal with the addition of a small quantity of Tin.
5. Sesquioxide of Iron. $Fe^2 O^3$.	No change.	Becomes black and magnetic.	When a small quantity is present, the glass is yellow while hot, but colorless when cold. If a large quantity is present, it is red when hot, yellow on cooling. When an excess is present, it is dark red when hot, dark yellow after cooling.	On Platinum wire the glass becomes bottle green. On charcoal with tin, the glass containing the oxide of iron becomes bottle green. On the platinum wire, the glass heated with Tin immediately appears copperas green.
6. Oxide of Cobalt. $Co O$.	No change.		Deep blue, while hot, and when cold. Should an excess be present, the glass will appear of a blackish hue.	
7. Oxide of Nickel. $Ni O$.	No change.		Strongly colored.—When a small quantity is present, the glass is violet when hot, and paler reddish brown when cold.	The glass containing the oxide becomes dull or quite opaque from the presence of finely divided metallic nickel. By long blowing, the particles of nickel adhere to one another, forming a globe or bead, and the tin becomes colorless.

With Microcosmic Salt on Platinum Wire.		With Carbonate of Soda on Charcoal or Platinum.	With a Solution of Nitrate of Cobalt, in the Oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
As with Borax ; but the color disappears entirely when cold.	The glass is colorless when hot and cold ; by this reaction it is distinguished from iron. The glass remains also clear when over saturated.	Not fused ; soda absorbed by the charcoal, the metal remaining upon the surface of the support, in the state of a white or lightish grey protoxide.	No reaction.
As with Borax.		Insoluble.	0
Ditto.		Insoluble. The soda is absorbed by the charcoal, and the oxide remains, having a grey color.	0
When a large quantity is added, the glass appears violet, but on the addition of more, it acquires a black tinge. If the salt contains much oxide, on being heated strongly, gas is evolved. Salt-petre assists in bringing out the color, when added.	The glass becomes instantly colorless, both on platinum wire and charcoal. After reduction, the glass becomes perfectly fluid.	In the oxidating flame, upon platinum wire or platinum foil, it fuses to a soluble transparent green mass, becoming opaque and bluish green, on cooling. On charcoal it cannot be reduced.	0
The glass containing a certain quantity becomes yellowish red. When cooling it appears first yellow, then greenish, and lastly colorless. If a very large portion is present the color will be dark red while hot, and brownish red on cooling, changing to dirty green, and, when perfectly cold, brownish red. The colors disappear on cooling, quicker than with borax.	The glass is not changed from the presence of a minute portion ; but if a large quantity is present, it is red when hot, yellow during refrigeration, then greenish, and when quite cold, reddish. With the addition of Tin, the glass is first green, and then the color is destroyed.	Insoluble. On charcoal it is reduced ; and gives, on being pulverized, a very metallic magnetic powder.	0
As with Borax, but the color, however, is not so intense. By transmitted light, the glass appears reddish. If a bright iron wire is inserted into the mass when in fusion, Cobalt is deposited.		On Platinum wire, in small quantities, it affords a transparent slightly red soluble mass, which is grey when cold. It is reduced on charcoal, giving a grey magnetic powder.	0
Melts to a reddish glass, which is yellow on cooling. If more is added, the glass assumes a brownish red when hot, and a reddish yellow when cold.	Upon Platinum wire, not altered. Treated on charcoal with tin, the glass is, at the commencement, translucent and grey, but by continued blowing, the Nickel deposits, leaving the glass colorless.	Insoluble. It is easily reduced upon charcoal, giving small white shining metallic particles, which, on being triturated, are readily attracted by the magnet.	0

TABLE II.—COMPORTMENT OF

METALLIC OXIDES.	Per Se, on Charcoal.		With Borax on the Platinum Wire.	
	In the Oxidating flame.	In the Reducing flame	In the Oxidating flame.	In the Reducing flame.
8. Oxide of Zinc. $Zn\ O.$	Yellow bead, while hot;—white, when cold. It does not fuse, but when very hot, gives out an intense light.	It is gradually dissipated over the charcoal, with white flakes of oxide of zinc.	Very soluble, forming a clear mass, which, if perfectly saturated, remains colorless when cold. By a strong heat, it is reduced, acting on the platinum.	The saturated mass will become enamel-white upon platinum wire. On charcoal the oxide is reduced, and the zinc volatilized. The charcoal also acquires a coating.
9. Oxide of Cadmium. $Cd\ O.$	No change, upon Platinum foil.	Disappears in a short time, covering the charcoal all over with a dark yellow powder, which is only clearly discerned when the mass cools.	When large quantities are present, the Borax melts to a clear yellowish glass; this color nearly disappears on cooling. When the borax is nearly saturated, the glass formed is sometimes, here and there, milk-white, and, when perfectly saturated, it becomes enamel white, if slowly cooled.	On charcoal the glass containing the oxide of Cadmium boils; the cadmium is reduced and volatilizes, and the charcoal becomes coated with dark brown oxide.
10. Oxide of Lead. $Pb\ O.$	Minium—red lead—when slightly heated, becomes black; but, upon increase of temperature, it is converted into a yellow oxide.	It is immediately reduced to a metallic globule, which gradually sublimes,—leaving a yellow residue.	Readily fuses to a clear yellow glass, colorless when cold. If a large quantity is present, the globule will be here and there opaque. With an excess, it is enamel-yellow when cold.	The glass diffuses itself over the charcoal: becomes turbid, and boils. The lead is reduced and the glass becomes again clear. It is very difficult to obtain, in the process, a bead of lead.
11. Teroxide of Bismuth. $Bi\ O_2.$	On Platinum foil, it fuses readily to a dark brown mass, which becomes pale yellow, on cooling.	On charcoal it is quickly reduced to grains of Bismuth,—which, with long blowing, vaporize, leaving the charcoal covered with yellow oxide.	Readily fusible to a limpid glass, which when a certain quantity is present, is yellow when hot, colorless when cold. A large quantity gives a yellowish red glass when hot,—while cooling, it is yellow, and when perfectly cold, opalescent.	On charcoal the glass becomes at the commencement grey and turbid:—boils, the oxide of bismuth being reduced; it again becomes perfectly clear. With Tin added, it is first grey, and then the whole of the bismuth separates, and the glass appears clear and limpid.
12. Sesquioxide of Uranium. $U_2\ O_3.$	It is converted into the protoxide: it blackens, but does not fuse.		As with oxide of Iron, but the color is somewhat clearer. When the glass is strongly saturated, it is streaked with enamel-yellow.	Gives the same color as oxide of Iron. The green glass, when saturated to a certain point, is blackened by an intermitting flame. It is either crystalline or enamel.
13. Oxide of Copper. $Cu\ O.$	Forms a black globule, which flows over the charcoal, and the under-surface is reduced.	By a temperature at which the copper does not fuse, the oxide is reduced, presenting the fine metallic lustre of the metal. When the blowing ceases, the surface of the metal is re-oxidised, and becomes black or brown. By increasing the heat, a globule of copper is obtained.	The glass is not very strongly colored. A small proportion of the Oxide gives a grass-green when hot, becoming blue on cooling. By a larger dose of the oxide, the glass is intensely green, appearing opaque; on cooling, it becomes opaque, and greenish blue.	At a certain point of saturation, the glass becomes colorless; but in a few moments it becomes red and opaque. By long blowing, the copper is precipitated in the metallic state upon charcoal, and the glass becomes colorless. When treated with Tin, the glass is opaque, as brownish red on cooling.

With Microcosmic Salt on Platinum Wire.		With Carbonate of Soda on Charcoal or Platinum.	With a Solution of Nitre of Cobalt, in the Oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
Like Borax, but with this difference, that the saturated glass becomes enamel white upon cooling.		Insoluble. Is reduced on charcoal, covering it with a sublimate of oxide. By a strong flame, the metal may be inflamed.	Gives a beautiful green color.
With large quantities, it fuses to a transparent glass; if saturated, it becomes milk-white on cooling.	Upon charcoal, Cadmium is slowly and imperfectly reduced; the metal is volatilized, and a very small quantity of a dark red oxide coats the support. This color, when cold, is a beautiful orange red.	Insoluble. On charcoal it is immediately reduced; the metal is converted into vapor, and the charcoal becomes coated with a dark yellow oxide.	0
As with Borax. It is necessary to add a large quantity of the oxide of Lead, to obtain a decided color when hot.	On charcoal the glass becomes gray and turbid. If an excess be present, the charcoal will be coated with a yellow oxide of lead. When Tin is added, the glass never becomes opaque, but a little more turbid, and of a darker grey.	In the oxidating flame, upon Platinum wire, it fuses readily to a limpid glass, which, upon cooling, becomes yellow and opaque. On charcoal the oxide is readily reduced.	0
A small quantity fuses to a transparent glass. If a large quantity is present, the glass will be yellow when hot, but colorless when cold. If a certain quantity is present, an enamel white here and there is often produced. When an excess is present, enamel-white on cooling.	On charcoal, particularly when Tin is added, the glass remains colorless and limpid while hot; but on cooling becomes opaque and greyish black.	Easily reduced upon charcoal. When volatilized, it gives no color to the flame, by which means it is distinguished from Antimony, Tellurium, & cetera.	0
Dissolves, giving a clear yellow glass, which becomes green on cooling.	The glass containing the oxide partakes of a beautiful green color, which on cooling becomes finer and clearer.	Insoluble. With a small addition of soda it gives indications of melting; with a greater quantity of soda it becomes yellowish brown; and by a still greater addition, the oxide is absorbed by the charcoal unaltered.	0
The glass containing the same quantity of oxide as with Borax, is not so highly colored. A small quantity of oxide gives a green glass, when hot; blue, when cold. A greater quantity gives a beautiful green, when hot; blue, when cold. An excess of oxide gives an opaque glass when hot; a greenish blue, when cold.	When nearly saturated, the glass is of an intensely dark green while hot, and on cooling appears opaque, and of a reddish brown. When the glass containing a very small quantity is treated with Tin, it is opaque and reddish brown when cooled.	It dissolves upon the Platinum wire to a limpid green glass, which, by refrigeration, loses its color, becoming opaque. It is readily reduced upon charcoal, and gives one or more beads of copper.	0

TABLE II.—COMPORTMENT OF

METALLIC OXIDES.	Per Se on Charcoal.		With Borax upon Platinum Wire.	
	In the Oxidating flame.	In the Reducing flame.	In the Oxidating flame.	In the Reducing flame.
14. Oxide of Silver. Ag O.	Reduced very rapidly.		Partly reduced, and partly dissolved. On cooling, the glass becomes milk-white or opaline, owing to unequal distribution of the dissolved oxide. Metallic silver melts with borax, giving the same reaction.	On charcoal the glass containing the oxide becomes grey, on account of the reduced silver. Postcedently, it becomes limpid and colorless, from the precipitation of the silver. This metal can be obtained as a bead.
15. Oxide of Mercury. Hg O.	Instantly reduced and volatilized. Should the whole not sublime, foreign matters will have been present, so that by this means adulteration is detected.		As with microcosmic salt.	
16. Bin oxide of Platinum. Pt O ₂ . 17. Bin oxide of Palladium. Pd O ₂ . 18. Sesquioxide, Rhodium. R ₂ O ₃ . 19. Bin oxide of Iridium. Ir O ₂ . 20. Oxide of Ruthenium.	Easily reduced; the metallic particles will not adhere to form a globule.		Reduced, but not soluble. The metallic particles alone upon charcoal do not form globules. These Metals have no action upon the Fluxes, —which can only serve to detect the foreign metals they may be combined with. They are best examined by cupellation with lead.	
21. Teroxide of Gold. Au O ₃ .	The instant the teroxide becomes red hot it is reduced.		It is not soluble, but is reduced, and can, by fusion, be obtained as a metallic globule. Gold has no tendency to combine with Oxygen or Sulphur, and hence retains its brilliancy in the open air for any length of time. It melts at 2016°; its density is 19.6; it is not acted on by any single acid, but is dissolved by aqua regia, and by a mixture of nitric and hydrofluoric acids.	
22. Bin oxide of Tin. Sn O ₂ .	The protoxide inflames like touch-wood, and is converted into the sesquioxide. The sesquioxide burns brilliantly, but it remains unchanged.	The sesquioxide of tin is reduced by long heating.	In small quantities it dissolves but very slowly, forming a glass which is colorless and limpid, whether hot or cold,—it may, perhaps, become opaque. A glass perfectly saturated, when heated, becomes opaque, loses its globular form, and appears a confused crystalline mass.	A glass containing a small quantity of the oxide is not altered. If more be added, the oxide is partly reduced upon charcoal.

With Microcosmic Salt on Platinum Wire.		With Carbonate of Soda.	With a Solution of Nitrate of Cobalt, in the Oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
<p>Metallic silver, as well as the oxide, gives a yellowish glass. If a large quantity is present, the glass becomes opaline, and appears yellowish by daylight, red by candle light.</p>	<p>As with Borax.</p>	<p>It is instantly reduced.</p>	<p>0</p>
<p>All the compounds of Mercury are volatile ; mixed with Tin or Iron Filings, and heated in a glass tube, metallic Mercury distills over.</p>		<p>When mixed with soda, and heated to redness in a closed tube, it is reduced and vaporized. The sublimate condenses in the coldest part of the tube, as a grey coating, which, when agitated, unites, forming a globule.</p>	<p>0</p>
<p>As with Borax.</p>			<p>0</p>
<p>As with Borax.</p>			<p>0</p>
<p>It dissolves in small quantities, very slowly, giving a limpid and colorless glass, which is not changed on cooling.</p>	<p>The glass containing the oxide is not altered, either upon Platinum wire or charcoal.</p>	<p>Upon the Platinum wire, Peroxide of Tin—stannic acid—effervesces, combining with the soda, forming an unmeltable tumacious mass. Upon charcoal, it is converted into the metallic state.</p>	<p>Assumes a bluish green color.</p>

TABLE II.—COMPORTMENT OF

METALLIC OXIDES, AND ACIDS.	On Charcoal without Reagents.		With Borax upon Platinum Wire.	
	In the Oxidating flame.	In the Reducing flame.	In the Oxidating flame.	In the Reducing flame.
23. Terioxide of Antimony. $Sb O^3$.	It is displaced without change, and deposited upon another part of the charcoal.	It is reduced, and sublimes. The charcoal becomes coated with a white oxide, and a greenish blue color is imparted to the flame. Antimony, when heated in a tube open at both ends, gives off large quantities of white vapors.	It dissolves in large quantities, and gives a limpid glass, which in the flame appears yellow, and on cooling becomes almost colorless. On charcoal, the dissolved oxide becomes paler: an addition of Tin does not alter this effect.	The glass, when treated only for a short time in the oxidating flame, becomes grey and cloudy, from particles of the reduced antimony: these are quickly volatilized, and the glass becomes lucid. When treated with Tin, the glass becomes ash-grey or black, whether there is a large or a small proportion of the oxide present.
24. Sesquioxide of Chromium. $Cr^3 O^3$.	Unchanged.		It colors the Borax highly, but it dissolves slowly. If an equal quantity is present, the glass will be yellow when hot; yellowish green when cold. If a larger quantity is present, it is dark red when hot; on cooling, yellow; and when cold, emerald green, with a trace of yellow.	The slightly saturated glass is of a beautiful green, when hot or cold. When a greater quantity is added, the color is more strongly marked, becoming emerald green. The addition of Tin does not produce any change.
25. Tellurous Acid. $Te O^3$.	Melts, and is reduced with effervescence. The reduced metal volatilizes, and covers the charcoal with a coating of white oxide.		Dissolves, forming a limpid colorless glass: heated upon charcoal, its color is grey, becoming opaque by reduction, from the metallic particles disseminated through the mass.	
26. Molybdic Acid. $Mo O^3$.	It melts, and is absorbed by the charcoal. In a strong reduction-flame, particles of the metal appear, which separate in the form of a grey metallic powder.		Soluble, forming a limpid glass, which appears yellow upon cooling, but is colorless when cold. When a large proportion of acid is present, the glass is dark yellow when hot; opaline when cold. With an excess, the glass is dark red when hot; becoming, on cooling, of an opalescent-bluish grey.	The glass, treated in the oxidating flame, with a very small quantity of acid, is colored brown. If a little more be added, the glass becomes wholly opaque.
27. Niobic Acid. $Nb O^3$.	Becomes greenish yellow in the heat; white when cold.		Like Tantalic acid.	
28. Pelopic Acid. $Pp O^3$.	Yellow when hot; white when cold.		Ditto.	

With Microcosmic Salt on Platinum Wire.		With Carbonate of Soda.	With a Solution of Nitrate of Cobalt, in the Oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
Dissolves with effervescence, giving a clear glass, which, when hot, appears of a feeble yellow color.	The saturated glass upon charcoal becomes milky at first, but in a short time clear; the antimony being reduced and dispersed in vapor. When treated with Tin, the glass is green; this color is owing to the reduced metal, but it soon becomes clear with strong blowing. The Tin produces always a murky-gray color, when a very small proportion of the oxide of antimony is present.	Upon charcoal, it is very easily reduced. The metal fumes and covers the charcoal with a coating of white oxide.	0
Soluble, forming a limpid glass, which is reddish when hot, and of a dull green color on cooling. When perfectly cold, it appears of a beautiful green hue.	As in the oxidating flame. The colorations appear more marked; this is the same under the influence of Tin.	In the oxidating flame, upon Platinum wire, it is dissolved with the formation of a dark brown yellowish glass, which, upon cooling, becomes yellow and opaque. In the reducing flame, the glass is opaque, and green when cold.	0
As with Borax.		Soluble on the Platinum wire, producing a limpid and colorless glass, which becomes white upon cooling. Upon charcoal, it is reduced, volatilizes, and leaves upon the support a coating of oxide.	0
Soluble to a clear glass, which, by a moderate addition of acid, is yellowish green, and on cooling, nearly colorless. Treated on charcoal, it becomes opaque, but, after cooling, of a fine green color.	The glass assumes a dark dirty green color, which, on cooling, changes to a fine clear green; this is analogous to the color presented by sesquioxide of chromium. Upon charcoal, its behaviour is similar. An addition of Tin has the effect of darkening this green tinge.	Fusible upon the Platinum wire, with effervescence, giving a clear glass, which, on cooling, becomes milk-white. When treated upon charcoal, the mass is absorbed, and a large quantity of the Molybdic acid is reduced. When levigated, the scoriae present the appearance of a steely-grey powder.	0
A large quantity gives a clear colorless glass.	First, violet; then a blue glass.	Like Tantalic acid.	A grayish glass while hot; dirty green when cold.
Ditto.	A brown glass. The presence of a little sulphate of iron makes the glass blood-red when hot.	Ditto.	Gives a light-grey color.

TABLE II.—COMPORTMENT

ACIDS.	On Charcoal without Reagents.		With Borax upon Platinum Wire.	
	In the Oxidating flame.	In the Reducing flame.	In the Oxidating flame.	In the Reducing flame.
29. Tungstic Acid. Wo O ³ .	Unchanged, if the flame is not a very strong one.	Blackens,—but does not fuse.	Dissolves readily to a limpid colorless glass. A great proportion of the acid gives a yellow when hot; and a greater quantity imparts to the glass the property of appearing opaque in the flame. If an excess be present, the glass becomes spontaneously enamel white, on cooling.	A small quantity of the acid does not alter the color of the glass. A greater proportion renders it yellow. A still greater quantity produces a dark yellow when hot, which is yellowish brown upon cooling. Tin darkens the colors, if the glass is not fully saturated.
30. Vanadic Acid. V O ³ .	Fusible.—The portions found in contact with the charcoal are reduced and absorbed. The others assume the color and brilliancy of graphite. They are the protoxide of Vanadium.		Soluble to a limpid glass, which, by a small quantity of this acid, is colorless; but, by a greater addition, becomes yellow.	The strongly yellow-colored glass changes, so that it appears brown when very hot; and, on cooling, of a beautiful chrome-green.
31. Tantallic or Columbic Acid. Ta O ³ .	Unchanged.		Soluble; forms a limpid colorless glass, which appears opaque in the flame. If more Tantallic acid be added, it appears enamel white on cooling.	As in the oxidating flame.
32. Titanic Acid. Ti O ³ .	Unchanged.		Readily soluble to a limpid colorless glass, which, by a greater addition of the acid, becomes yellow, and is colorless after cooling. At a certain degree of saturation the glass appears streaked with enamel-white; if an excess is present, it becomes opaque on cooling.	A small proportion of the acid colors the glass yellow, while a larger quantity gives a dark yellow or brown shade. A glass when saturated appears enamel blue in the flame.

¶ *Description of the Phenomena presented by the sublimates of some Metals, when melted or heated strongly upon Charcoal before the Blowpipe flame.*

Selenium.—This element affords a shining steel-grey slightly metallic deposit, which presents a violet, but when in thin layers, a blue, lustre. In the oxidating flame it readily changes its place

With Microcosmic Salt on Platinum Wire.		With Carbonate of Soda.	With a Solution of Nitrate of Cobalt, in the Oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
Soluble, producing a clear glass; if more strongly saturated, the glass appears yellow while hot.	A beautiful clear blue glass is obtained. If the tungstic acid is combined with iron, the color will be blood-red. The glass containing the iron is colored blue when tin is added, and green if there is a considerable portion of iron.	Soluble, upon the Platinum wire, forming a dark yellow glass, which, when cooling, becomes crystalline and opaque, with a tinge of white or yellow. Upon charcoal, with a small quantity of soda, a large quantity of the acid is reduced; by a greater addition of soda, the whole is absorbed by the charcoal. A conjunction of the shining metal, and the yellow oxide of tungsten, is often obtained with the soda.	0
As with Borax.		It fuses with the soda, and is absorbed by the charcoal.	0
Dissolves readily and easily in large quantities, forming a colorless glass, which cannot be rendered opaque in the flame, but which becomes spontaneously so upon cooling.	As in the oxidating flame.	It combines with the soda, effervescence taking place, but it is neither fused nor reduced.	0
Dissolves easily, forming a limpid glass, which, when containing a certain proportion of acid, is yellow when hot, and colorless upon cooling. The glass, treated in the oxidating flame, presents the following appearances: when hot, the color is yellow, becoming, on cooling, red; and after, assumes a beautiful violet tinge. When a greater quantity of the acid is added, the color produced is so intense that the glass appears opaque, but has not an enamel aspect. If the metal contains iron, the glass, upon cooling, will be brownish yellow, yellow, or brownish red. An addition of tin destroys this color produced by the iron, and the glass becomes violet.		Soluble, forming, after the effervescence and decrepitation has taken place, a dark yellow glass. The glass is not absorbed by the charcoal; after cooling, it appears greyish white, or white. If the glass is heated red hot, it crystallizes, and develops so much caloric that the bead spontaneously attains a white heat. The titanio acid is not reduced.	It is colored black, or greyish black.

upon the charcoal, and in the reducing flame the color is bright blue. All the salts of selenious acid, heated on charcoal before the Blowpipe, impart a magnificent blue color to the flame, emitting the unmistakable and offensive smell of foul horseradish.

Tellurium.—Covers the charcoal with an oxide, at the same

distance from the assay as selenium ; the deposit is white, with a red or dark yellow border, easily removable by the oxidating, and driven off by the reducing flame, producing a green, but, when selenium is present, a bluish-green appearance.

Arsenic.—The charcoal is covered with arsenious acid, which is white,—in thin layers, greyish, from the charcoal being seen through it,—and is deposited at a long distance from the assay. It flies off, when treated in either flame.

Antimony.—This element covers the charcoal with an oxide which is white, in thin laminæ, bluish, and which is deposited at a shorter distance from the assay than the arsenical vapor ; when treated in the oxidating flame, it is driven from place to place unaltered, but in the reducing flame it changes its position, producing a feeble bluish-green color. The antimonial sublimate is not so volatile as the arsenical, so that by this means they can be readily distinguished.

Bismuth.—Covers the charcoal with an oxide. The sublimate is dark orange-yellow while hot, lemon-yellow when cold, and when in thin layers, bluish ; it is deposited not nearly so far from the assay as the antimonial sublimate ; it can be driven from place to place either by the oxidating or the reducing flame. It gradually diminishes, a portion of it being volatilized.

Lead.—This metal covers the charcoal with an oxide at the same distance from the assay as bismuth. The sublimate is, while hot, of a dark citron-yellow, upon cooling, sulphur-yellow, and when in thin laminæ, bluish ; in other respects, it behaves like the sublimate from bismuth.

Cadmium.—Coats the charcoal with an oxide, at the same distance from the assay as bismuth. The sublimate is best seen when cold ; its color is reddish-brown, in thin films yellow ; it can be volatilized in either flame.

Zinc.—The charcoal is covered with oxide, which is not so far from the assay as that of bismuth or lead. When hot, it is yellow, and strongly phosphorescent; when cold, nearly white. It does not volatilize in the oxidating flame. A drop of nitrate of cobalt added to it, in the oxidating flame, colors it beautifully green.

Tin.—Deposits an oxide upon the charcoal, which is feebly yellow, and moderately phosphorescent when hot; on cooling it is white, and almost touches the assay. It assumes, with cobalt solution, a bluish-green color, which may be readily distinguished from that produced by oxide of zinc.

Silver.—If heated alone in the oxidating flame, it covers the charcoal with a slight dark-red precipitate. In combination with a little lead, it produces at first a yellow sublimate of oxide of lead; but when the silver becomes more minutely disseminated, a dark red sublimate appears beyond the yellow oxide of lead. If the silver contains a little antimony without lead, only a slight red sublimate is obtained. If it contains both lead and antimony, it assumes a carmine red color, after the greater part of these metals are volatilized.

Iodine, Bromine, Chlorine, and Sulphur.—Iodine vapors may be distinguished from those of Indigo by the irritation the former cause when inhaled. Indigo gives purplish fumes, which smell like anilin. In examinations with the Blowpipe, the operator does not always encounter the pure substances, which are recognized by the characteristic sublimates they yield, when heated upon charcoal,—but sometimes meets with combinations which deposit a white sublimate, admitting of being driven from place to place in the oxidating and reducing flames, thus resembling oxides of tellurium or antimony; these are particularly the following:—sulphides of potassium and sodium; chlorides of potassium, sodium, ammonium, lead, and mercury; bromides of potassium and sodium; and iodides of potassium and sodium. The most volatile of these combinations are the chlorides of

ammonium, mercury, and lead; the first two volatilize without melting, the others require to be strongly treated in the reducing flame, and the majority of them are absorbed by the charcoal before sublimation takes place. The sublimates from the most volatile are generally deposited at some distance from the assay, while those of the less volatile lie nearer the assay, and are more difficultly displaced by the flame. These sublimates are distinguishable from those of the oxides of tellurium and antimony, inasmuch as they disappear in the reducing flame without producing any color.

¶ *Qualitative Examination of Minerals, Ores, and the Products of Metallurgic Operations for Metallic and Non-metallic Bodies before the Blowpipe.*

a. EXAMINATION OF THE METALLIC OXIDES WHICH FORM ALKALIES AND EARTHS.

§ 1. POTASSA—K O—*Presence in the Mineral Kingdom.*

Potassa is found in combination with acids only, as for example :—

a. With Sulphuric Acid, as well *per se*, as in combination with Earths and Water, namely, *per se*, in *Sulphate of Potassa* [K O, S O³]; with Sulphate of Alumina, in *Alum* [K O, S O³ + Al² O³, 3 S O³ + 24 aq]; and with Sulphates of Lime and Magnesia, in *Potassa-Pollyhallite* [K O, S O³ + Mg O, S O³ + 2 (Ca O, S O³) + 2 aq.]

b. With Nitric Acid, in *Saltpetre* [K O, N O²], but generally in combination with Sulphate and Carbonate of Lime, and Chloride of Calcium; and,

c. With Silicic Acid, in various silicates of different bases, as for example, in *Felspar* [K O, Si O² + Al² O³, 3 Si O²]; *Leucite* [3 K O, 2 Si O² + 3 Al² O³, 2 Si O²]; *Apophyllite* [K O, 2 Si O² + 8 (Ca O, Si O²) + 16 aq], *Potassa-Tourmaline*—*Schorl*;—and in some micaceous minerals, *et cetera*. Potassa is found also

in Murchisonite, Raphilite, Chrom-mica, Diploite, Nuttallite, Pinite, Elaeolite, Nepheline, Ryakolite, Couzeranite, Weissite, Tachylite, Humboldtite, Agalmatolite, Rosite, Polyargite, Zeagonite, Antrimolite, Gigantolite, Chabasite, Gmelinite, Phakolite, Pyrrargillite.

Examination for Potassa.

The easily-fusible salts of potassa, with the exception of the phosphate and borate, are recognized when held in the points of the forceps, no soda being present, by the intense violet color given to the outer flame.

If the mineral contains a small quantity only of potassa, no coloring will ensue, unless the splinter of it is held properly in the apex of the flame; great care must also be taken to have the assay operated upon, free from all extraneous matters.

When soda or lithia is also an ingredient of the mineral, this mode is not applicable, as the flame will be colored yellow by the former, and crimson by the latter. HARKORT'S method for the determination of potassa, when the quantity of this alkali is not too small, is as follows:—Oxide of nickel is dissolved in borax, until the glass, upon cooling, assumes a dark brown color; to this glass is then added a portion of the substance under examination, and the mixture is treated in the oxidating flame; if the glass upon cooling retains its brown color, potassa is either absent or in too minute a quantity to be detected; but if, on the other hand, the bead acquires a bluish tint, similar to a diluted solution of oxide of nickel in ammonia, it indicates the presence of this alkali. LAMPADIUS observes, that the potassa and oxide of nickel glass is blue, while the oxide of nickel and soda glass is brown.

When substances, for instance native silicates, contain only traces of potassa, the above method cannot with certainty be resorted to; therefore, the moist way must also be applied, as subsequently described: 50 to 75 milligrammes of the fine powdered body are mixed with twice their volume of soda, and pressed into a cavity in a plate of charcoal, and smelted in the oxidating flame: if much magnesia or lime is present, the assay will fuse with difficulty. The ignited mass is now removed from the sup-

port with the platinum forceps, and any adhering carbonaceous matter burnt away. The mass is pulverized between paper in an agate mortar, then treated in a porcelain basin with hydrochloric acid, and evaporated to dryness over the flame of the lamp. Water is now added, to dissolve the formed metallic chlorides, and, after some time, the solution is poured from the undissolved silica into another basin. This liquid is evaporated until it becomes highly concentrated, and then a few drops of an alcoholic solution of chloride of platinum and sodium are added. If potassa is present, the double salt of chloride of platinum and potassium, which is insoluble in alcohol, will precipitate as a lemon-yellow powder. If there is only a small quantity of alkali present, the precipitate will not be perfectly visible for some minutes. The presence of organic matters does not prevent this reaction.

In combinations of sulphuric acid, which contain both potassa and soda,—as, for instance, Polyhallite,—it is only requisite to reduce on charcoal the sulphate to a sulphide, and treat with hydrochloric acid, water, *et cetera*, as in the previous example.

By the preceding methods, one per cent. of potassa in silicates, and other substances, can be recognized.

BERZELIUS has given a similar examination, in one of his treatises upon the quantitative separation of potassa from soda.

§ 2. SODA—Na O—*Presence in the Mineral Kingdom.*

Soda is never found *per se*, but always in combination, as,—

a. With Chlorine, in *Rock Salt* [Na Cl], at times containing traces of chloride of ammonium.

b. With Fluorine and Fluoride of Aluminum, in *Kryolite* [$3 \text{ (Na Fl)} + \text{Al}^3 \text{ Fl}^3$];

c. With Carbonic Acid and Water, in *Natron* [$\text{Na O, C O}^2 + 10 \text{ aq}$]: and *Trona—Urao*—[$2 \text{ Na O, } 3 \text{ C O}^2 + 4 \text{ aq}$]; also with Carbonic Acid, Carbonate of Lime, and Water, in *Gaylussite* [$\text{Ca O, C O}^2 + \text{Na O, C O}^2 + 6 \text{ aq}$];

d. With Sulphuric Acid, in *Glauber Salt* [$\text{Na O, S O}^3 + 10 \text{ aq}$], which is often contaminated with carbonate of soda, chloride of sodium, and chloride of calcium;

e. With Sulphuric Acid and Sulphate of Lime, in *Glauberite* [$\text{Na O, S O}^3 + \text{Ca O, S O}^3$];

f. With Nitric Acid, in *Nitrate of Soda* [Na O, N O^5];

g. With Boracic Acid and Water, in *Borax—Tincal*— $[\text{Na O, 2 B O}^3 + 10 \text{ aq}]$; and,

h. With Silicic Acid, in various Silicates,—as, for example, in *Tetartine* or *Albite*—*Soda Felspar*— $[\text{Na O, Si O}^2 + \text{Al}^2 \text{ O}^3, 3 \text{ Si O}^2]$; in *Sodalite* from Vesuvius $[\text{Na Cl} + 2 (\text{Al}^2 \text{ O}^3) + 2 (3 \text{ Na O, Si O}^2) + 2 (\text{Al}^2 \text{ O}^3, \text{Si O}^2)]$; in *Achmite* $[3 (\text{Na O, Si O}^2) + 2 \text{ Fe}^2 \text{ O}^3, \text{Si O}^2]$; in *Soda Spodumene*—*Oligoklas*— $[3 (\text{Na O, Si O}^2) + 4 \text{ Al}^2 \text{ O}^3, 2 \text{ Si O}^2]$; in *Nepheline* $[\frac{2}{3} \text{ Na O}, \frac{1}{3} \text{ K O}] \text{ Si O}^2 + 2 (\text{Al}^2 \text{ O}^3, \text{Si O}^2)]$; in *Labradorite* $[\text{Na O, Si O}^2 + \text{Al}^2 \text{ O}^3, \text{Si O}^2 + 3 (\text{Ca O, Si O}^2 + \text{Al}^2 \text{ O}^3, \text{Si O}^2)]$; in *Soda Tourmaline*; *Analcime* $[3 \text{ Na O, 2 Si O}^2 + 3 (\text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2) + 6 \text{ aq}]$. This alkali occurs also in Arvedsonite, Bytownite, Andesine, Saussurite, Glaukolite, Indianite, Humboldtite, Retinalite, Brevicite, Chabasite, Gmelinite, Harringtonite, Thomsonite, and Comptonite, Fahlunite, Pechstein, Epistilbite, Wöhlerite, and with potassa in numerous other minerals.

Examination for Soda.

When a soda compound is smelted in the apex of the blue flame, the soda is readily recognized, from the reddish-yellow color given to the outer flame. The flame partakes of this color, but in a less degree, even when the body contains a large quantity of potassa or lithia. According to VON KOBELL, when chloride of potassium is mixed with one twenty-fifth or one thirtieth part of chloride of sodium, only the soda reaction is given.

The splinter from the soda mineral is held like that of potassa, in the points of the platinum forceps, when the soda reaction ensues.

§ 3. *LITHIA*— Li O —*Presence in the Mineral Kingdom.*

This alkali is found always in combination, as in,—

a. *Amblygonite*, which is a Phosphate of Alumina and Lithia,

mixed sometimes with a fluorine compound; its formula is $[2 \text{ Li O}, \text{ P O}^5 + 4 \text{ Al}^2 \text{ O}^3, 3 \text{ P O}^5]$;

b. Many Silicates of different bases: as, for example, in *Petalite* $[\frac{3}{2} \text{ Li}_2 \text{ O}] \cdot 4 \text{ Si O}^2 + 4 (\text{Al}^2 \text{ O}^3, 4 \text{ Si O}^2)$, it contains also hydrofluoric acid; *Lithion Spodumene* $[3 \text{ Li O}, 2 \text{ Si O}^2 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2]$; *Tourmaline*, *Lepidolite*, and many other Micas which contain hydrofluoric acid.

Examination for Lithia.

Compounds of lithia, when heated in the apex of the blue flame, upon the platinum wire, tinge the outer flame crimson. This characteristic appearance is best shown with the chloride of lithium. When the chloride is mixed with a salt of potassa, the red color alone is produced. When, on the contrary, the salt of lithia is contaminated with a salt of soda, then the reaction of soda only is observable; and this is also the case when a lithia salt contains both potassa and soda salts.

Lithion minerals, as, for example, Lithion-mica from Altenberg and Zinwald, give, when heated in the apex of the blue flame, an intense carmine color; but if the alkali is in very small quantities only, the reaction will be indistinct.

TURNER has given the following method for detecting traces of lithia in silicious minerals:—Knead into a paste, with water, one part of fluor spar, one and a half part of bisulphate of potassa, and a portion of the fine powdered mineral for investigation; then smelt the mixture in the ring of the platinum wire, in the apex of the blue flame. If the body contains lithia, the outer flame will assume a crimson tinge; if not, it will be colored violet, from the presence of potassa. MERLET employs, for this test, one part of the fine powdered mineral, and two parts of the mixture of fluor spar and bisulphate of potassa. The Amblygonite from Chursdorf, in Saxony, which contains eleven per cent. of lithia, gives to the outer flame an intense carmine color, provided that soda is absent. If soda is present, the outer flame will be colored more yellow than red. With bisulphate of potassa and fluor spar, if only traces of lithia are contained

therein, the red reaction will be given ; but here, if soda is also present, its reaction will prevail.

§ 4. *AMMONIA*— N H^3 —*Presence in the Mineral Kingdom.*

Ammonia is not met with in a free state, but in combination with chlorine and acids only, namely,—

a. With Chlorine, in native *Chloride of Ammonium* [N H^4 , Cl].

b. With Sulphuric Acid, in *Mascagnin*—Sulphate of Ammonia— $[\text{N H}^4 \text{ O}, \text{S O}^3 + \text{aq}]$; and,

c. With Sulphuric Acid, Alumina, and Water, in *Ammonalum* [$\text{N H}^4 \text{ O}, \text{S O}^3 + \text{Al}^2 \text{ O}^3, 3 \text{ S O}^3 + 24 \text{ aq.}$] Ammonia is found in small quantity in many other mineral substances, *exempli gratia*, in Tachylite, Rock Salt, Pikrosmine, Sassoline ; also in various aluminous and ferruginous minerals eviscerating organic matter.

Examination for Ammonia.

The ammoniacal salts are very easily recognized, by treating them in a glass tube, mixed with carbonates of soda, potassa, or lime, over a spirit-lamp. Carbonate of ammonia sublimes, which is known by its odor, and also by its coloring reddened litmus-paper blue. Its presence is infallibly shown by holding over the mouth of the tube, a glass rod moistened with hydrochloric acid, for if a trace only of ammonia is eliminated, a white cloud will be produced. When dry salts of ammonia are triturated in an agate mortar, or heated in a test tube with the caustic alkalies, or alkaline earths, the well-known penetrating odor of ammonia becomes immediately perceptible.

None of the ammoniacal compounds can sustain an incipient red heat, without being volatilized or decomposed. This arises from the gaseous nature of the alkali.

§ 5. *BARYTA*— Ba O —*Presence in the Mineral Kingdom.*

Baryta is found :—

a. As a Carbonate, in *Witherite* [$\text{Ba O}, \text{CO}^2$] ; and, in com-

bination with Carbonate of Lime and Manganese, in *Baryto-calcite* [$4 (\text{Ba O}, \text{C O}^2) + 4 (\text{Ca O}, \text{Co}^2) + \text{Mn O}, \text{C O}^2$];

b. As a sulphate, in *Heavy Spar*—Sulphate of Baryta— $[\text{Ba O}, \text{S O}^3]$; and in combination with Sulphate of Lime, in *Calcareous Heavy Spar* $[\text{Ba O}, \text{S O}^3 + \text{Ca O}, \text{S O}^3]$;

c. In Silicic Acid combinations, containing Baryta, Alumina, and also Water,—as in *Baryta Harmotome* $[3 \text{Ba O}, 2 \text{Si O}^2 + 5 (\text{Al}^2 \text{O}^3, 2 \text{Si O}^2) + 24 \text{aq}]$; and in a Silicic Acid compound containing Baryta, Strontia, Alumina, and Water, as *Brewsterite* $[3 (\text{Sr O} \text{ } \frac{\text{O}}{\text{Ba O}}) \text{Si O}^3 + 4 (\text{Al}^3 \text{O}^3, 3 \text{Si O}^2) + 18 \text{aq}]$.

Baryta occurs also in Dreelite, Psilomelane, Braunite, and Hausmannite.

Heavy Spar sometimes forms a constituent in metallic gangues; it is therefore necessary to examine for Baryta in the Ores, and also in the Slags produced in smelting them.

Examination for Baryta.

a. Witherite is readily recognized before the Blowpipe, as it behaves like pure Baryta.

Baryto-calcite, *per se*, is quite infusible. It becomes, however, when strongly ignited, alkaline. A sample from Cumberland, when strongly heated in the reducing flame, and moistened, gave no stain to silver.

It dissolves, with tumefaction, in borax, and forms a colored glass like manganese, which, in the reducing flame, becomes diaphanous.

It is dissolved in microcosmic salt, under powerful vesiculation, to a pellucid glass, which, if more strongly saturated, assumes the manganesian color. It is transparent in the reducing flame, and opaque when cold.

When heated with carbonate of soda, it is decomposed; the baryta and soda are absorbed by the charcoal, and the lime and protoxide of manganese remain behind.

b. Heavy Spar, preferable in a powdered state, as it decrepitates very strongly, when heated upon charcoal in the reducing flame, is converted into sulphide of barium, which gives off, when moistened, an hepatic smell, and possesses an astringent taste.

With soda, it melts to a pellucid limpid glass, which boils when strongly heated, and becomes absorbed as a strong hepatic mass, by the charcoal.

As Celestine—Sulphate of Strontia—behaves in a similar manner, the assay, in order to ascertain which is present, must be treated with hydrochloric acid and alcohol, as will be described under the head of Strontia. The chloride of barium does not give a colored flame. The Heavy Spar is distinguished from Celestine by the former, *per se*, being difficultly fusible, while the latter can readily be melted to a globule. From Celestine it is also distinguished by coloring the outer flame yellowish, when held with a forceps in the apex of the blue flame.

Calcareous Heavy Spar, *per se*, and with carbonate of soda, behaves like Heavy Spar; with this difference, that when heated with soda, the lime is not likewise absorbed by the charcoal, but remains as an infusible mass. This phenomenon is better remarked during the ignition, the lime running to various parts of the charcoal, and appearing slightly phosphorescent.

c. In Baryta Harmotome, and in Brewsterite, the baryta can neither be distinguished alone nor with glacial fluxes. It is therefore necessary to proceed as follows:—Smelt one part of the powdered sample, with one part of soda, and one part of borax, upon charcoal, to a bead. Pulverize the smelted assay, treat with hydrochloric acid, evaporate to dryness, and dissolve in an excess of water. Filter to separate the silica, and add to the filtrate diluted sulphuric acid, or bisulphate of potassa, to precipitate the baryta and strontia. Filter, wash, and reduce the residue to sulphides of barium and strontium, then treat with hydrochloric acid and alcohol, to determine whether both are present. If the alcohol flame be colored red, strontium is present. The alumina, which is not precipitated by bisulphate of potassa, or diluted sulphuric acid, may be separated from the solution in the manner described at page 127.

In assorted ores and slags, baryta cannot be detected by the Blowpipe alone; it is, therefore, always convenient to have recourse to the moist way. Such substances, after previously roasting those which contain arsenic or metallic sulphides, must

be melted, according to page 77, with one part of carbonate of soda and one part of borax ; and, if some difficultly reducible metals are present, 200 or 300 milligrammes proof lead, or 80 to 100 milligrammes of fine silver, must be added. The fused assay is treated with hydrochloric acid and water,—see page 79. To this liquid, from which the silica has been separated by filtration, bisulphate of potassa or diluted sulphuric acid is added, to precipitate the baryta. According to this method, it can be confounded with sulphates of strontia and lime only. But if the substance is very dilute, sulphates of baryta and strontia only are precipitated, as sulphate of lime is soluble in an excess of water. The precipitate is now affused on the filter with water, and dried ; if baryta only is present, after treating the residue left on the filter with hydrochloric acid and alcohol, and igniting, no red color will be imparted to the flame of the spirit. If colored red, the two earths may be contained therein. The other ingredients in the solution are detected, as stated under Lime, Magnesia, and Alumina, in their silicic acid combinations.

§ 6. *STRONTIA*—Sr O.—*Presence in the Mineral Kingdom.*

a. Strontia occurs as a Carbonate in *Strontianite* [Sr O, C O²] ; it is also a very small ingredient of *Arragonite*—*Carbonate of Lime*,—and, with Sulphate of Baryta and a trace of Carbonate of Lime, in *Stromnite*.

b. As a Sulphate, in *Celestine*, [Sr O, S O³] ; and sometimes in *Heavy Spar*.

c. In a Silicic Acid combination of Baryta, Strontia, Alumina, and Water, as *Brewsterite*. If such minerals occur in metalliferous gangues, a very minute quantity of Strontia may be detected in the dressed ores, and in the slags produced therefrom.

Examination for Strontia.

Strontianite alone, and with fluxes, comports itself before the Blowpipe flame, like the pure earth. If dissolved in hydrochloric acid, and the solution evaporated slowly to dryness, the dried

residue then burned with alcohol, the intense characteristic carmine color will be visible.

The pyrognostic properties of Arragonite will be mentioned under Lime.

Stromnite is most readily determined as follows:—The pulverized sample is treated on charcoal in the reducing flame, and the presence of strontia afterwards recognized by the tint communicated to the flame of alcohol, as previously stated.

Celestine fuses to an opalescent glass, and gives a reddish color to the outer flame when heated in a pair of forceps. When the glass is treated on charcoal, the sulphate of strontia is reduced to a sulphide, and when moistened, behaves towards silver like sulphide of barium. Celestine with soda behaves like Heavy Spar. When treated like Strontianite, it imparts a beautiful crimson hue to the alcoholic flame.

The casual presence of Strontia in Heavy Spar, is determined by reducing to sulphides, and treating with hydrochloric acid and alcohol; the strontia is recognized by the color imparted to the spirit when inflamed.

When Chloride of Strontium is treated on the ring of the platinum wire, in the apex of the blue flame, the whole flame is immediately perceived of a deep crimson; however, when the assay is fused, the red color vanishes, and thus the chloride of strontium is distinguishable from the chloride of lithium.

CHAPMAN, in the *Chemical Gazette*, vol. viii. page 428, gives the following as the Blowpipe reactions of baryta and strontia.

Chloride of barium, when mixed with chloride of strontium, in much less quantity than 50 per cent. of the former, entirely prevents the strontia flame from being rendered visible; and the accidental presence of soda can have nothing to do with this reaction, because if a mixture of even equal parts of chloride of barium and carbonate of soda is made, and the mass exposed before the blowpipe on a loop of platinum wire, the yellow flame derived from the soda will only prevail for a few minutes, and will then give place to the pale green flame of baryta, the whole of the soda being got rid of by rapid volatilization under the form of chloride of sodium. In like manner a mixture of chloride of

strontium, and carbonate of soda, will give, after a well continued blast, the characteristic flame of the former. * * * * Both the natural sulphate and the carbonate—the celestine and strontianite—produce, *per se*, a well marked crimson coloration, if not on the first application of the flame, at least after blowing for a few instants. It is true, that with previously ignited chloride of strontium, the flame is at first scarcely colored; but if the fused bead be shaken from the loop, and the platinum wire be again presented to the flame, the coloration is most intense and persistent; a phenomenon not easily explained, unless it be that the heat required to produce the reaction without the presence of water can scarcely be attained with the larger globule.

Thus much for this authority; his remarks, however, do not bear the slightest approximation with the true results.

Mr. E. E. HOLDEN, at the request of the English author, repeated some experiments on the subject, previously performed by the latter, and obtained similar reactions to those which had before been gained, as under:—

The presence of chloride of barium, when mixed with chloride of strontium, does *not* prevent the appearance of the red color in the Blowpipe flame.

Caustic strontia, dry or moist, has not the slightest characteristic effect, as to coloration, before the Blowpipe, and only its moist soluble salts impart a crimson tinge to the flame. Artificial sulphate, phosphate, and carbonate of strontia do not, under any circumstances, color the apex of the flame. When chloride of barium and nitrate of strontia are mixed in equal quantities, the strontia reaction predominates. A compound of equal weights of acetate of baryta and nitrate of strontia, deflagrates, and yields a greenish yellow flame. Desiccated acetate of strontia gives no characteristic coloration, but when kneaded into a paste with water, it imparts to the apex of the flame a most brilliant crimson. When the chlorides of barium, strontium, and calcium are incorporated, the carmine flame is alone perceptible.

Substances which neither color the outer Blowpipe flame nor the flame of alcohol, such as Brewsterite, assorted ores, slags, *et cetera*, must be transposed to a state in which the strontia is

separated from the other ingredients, and then investigated. The procedure is conducted in a manner similar to that given under baryta. When chloride of strontium contains chloride of calcium, it can be discovered by treating the mixture with carbonate of soda upon charcoal. The strontia, and any baryta, are absorbed, while the lime remains upon the surface of the support as an infusible mass.

Chloride of calcium, when heated on the ear of the platinum wire, communicates a red tinge to the outer flame. The color, however, is more feeble with the lime salt than with the chloride of strontium. A solution of sulphate of lime precipitates immediately salts of baryta, but not of strontia. The sulphate of lime is perfectly soluble in hydrochloric acid, which distinguishes it from the sulphates of baryta and strontia. Chromate of potassa readily gives a yellowish precipitate of chromate of baryta, with solutions of the salts of this earth. The precipitate is *insoluble in chloride of ammonium*.

§ 7. LIME—Ca O.—*Presence in the Mineral Kingdom.*

Lime is met with—

- a. As Fluoride of Calcium, in *Fluor Spar* [Ca Fl] ;
- b. As Carbonate of Lime, [Ca O, C O²] in *Chalk, Limestone, Calcareous Spar, Kalk Tuff—Tufa, Rock Milk—Berg-milch, Schiefer Spar—Slate Spar, Anthraconite, Stinkstone*—from the strongly fetid odor it exhales when slightly rubbed—*Marl*, and in bituminous marly slate ; further, with a trace of Carbonate of Strontia, in *Arragonite*, also with carbonic acid, in *Dolomite*, [Ca O, C O² + 3 (Mg O, C O²)] *et cetera*, and with Carbonate of Baryta, in *Barytocalcite* ;
- c. As Sulphate, in *Gypsum—Selenite*—[Ca O, S O³ + 2 aq], and *Anhydrite* [Ca O, S O³] ; further, with Sulphate of Potassa and Sulphate of Magnesia, in *Polyhallite* ; with Sulphate of Soda, in *Glauberite*, and with Sulphate of Baryta, in *Calcareous Heavy Spar* ;
- d. As Phosphate of Lime, with Chloride of Calcium,—the

latter being sometimes more or less replaced by Fluoride of Calcium,—in *Apatite*—so named by WERNER, from *anaraw*, to deceive, in allusion to its being easily mistaken for certain other minerals,— $[\text{Ca} \overset{\text{Cl}}{\text{F}}] + 3 (3 \text{ Ca O}, \text{P O}^5)$]; or, $[\text{Ca Cl} + 3 (3 \text{ Ca O}, \text{P O}^5)] = [\text{Ca Fl} + 3 (3 \text{ Ca O}, \text{P O}^5)]$;

e. As Arseniate of Lime, in *Pharmacolite*—from the Greek, in allusion to its containing a poisonous ingredient— $[2 \text{ Ca O}, \text{As O}^5 + 6 \text{ aq}]$, and *Hardingerite* $[2 \text{ Ca O}, \text{As O}^5 + 4 \text{ aq}]$;

f. As Tungstate of Lime, in *Schuerstein* $[\text{Ca O}, \text{W O}^3]$;

g. As Titanite and Silicate of Lime, in *Titanite* and *Sphene*—brown and yellow *Menaccanite*— $[\text{Ca O}, 3 \text{ Ti O}^2 + \text{Ca O}, 2 \text{ Si O}^2]$, and $[\text{Ca O}, 2 \text{ Ti O}^2 + 2 (\text{Ca O}, \text{Si O}^2)]$;

h. As Tribasic Tantalate of Lime and Yttria, containing traces of proto-tungstate of iron, lime, and oxide of uranium, in Dark *Ytthro-tantalite* $[\overset{3}{3} \overset{\text{Y}}{\text{Ca}} \overset{\text{O}}{\text{O}}] + \text{Ta O}^3$]; and with a considerable quantity of Tribasic proto-tungstate of iron, in Black *Ytthro-tantalite* $[\overset{3}{3} \overset{\text{Y}}{\text{Ca}} \overset{\text{O}}{\text{O}}] + \{\overset{\text{Ta}}{\text{W}} \overset{\text{O}^3}{\text{O}^3}\}$: the Yellow *Ytthro-tantalite* containing a large proportion of peroxide of uranium, its formula is $[3 \text{ Y O } \{\overset{\text{Ta}}{\text{U}} \overset{\text{O}^3}{\text{O}^3}\}]$;

i. As neutral and bibasic Borate of Lime, with Bisilicate of Lime, in *Datolite* $[\text{Ca O}, 2 \text{ B O}^3 + \text{Ca O}, 2 \text{ Si O}^2 + \text{aq}]$, and *Botryolite*,—from the Greek, owing to the resemblance in form to grapes— $[2 (2 \text{ Ca O}, 2 \text{ B O}^3) + 2 (\text{Ca O}, 2 \text{ Si O}^2) + 2 \text{ aq}]$;

k. In Silicates; and indeed:—

¹ In a Silicate where the Lime alone is in combination with the acid, namely, in *Tabular Spar*—*Wollastonite*— $[3 \text{ Ca O}, 2 \text{ Si O}^2]$;

² In Silicates of Potassa and Lime, with Water,—for example, *Apoklas*—*Apophyllite*— $[9 \{\overset{\text{Ca}}{\text{K}} \overset{\text{O}}{\text{O}}\} \text{Si O}^2 + 16 \text{ aq}]$; which generally contains hydrofluoric acid;

³ In Silicate of Lime, or Lime and Alkalies, with Silicate of Alumina and water of crystallization; *exempli gratia*, in—*Stilbite* $[3 \text{ Ca O}, \text{Si O}^2 + 3 (\text{Al}^3 \text{ O}^3, \text{Si O}^2) + 18 \text{ aq}]$; *Desmine* $[\overset{\text{Na}}{\text{Ca}} \overset{\text{O}}{\text{O}}] 3 \text{ Si O}^2 + 3 (\text{Al}^2 \text{ O}^3, 3 \text{ Si O}^2) + 6 \text{ aq}]$; *Heulandite* $[3 (\text{Ca O}, \text{Si O}^2) + 4 (\text{Al}^2 \text{ O}^3, 3 \text{ Si O}^2 + 18 \text{ aq})]$;

Laumonite $[3 \text{ Ca O}, 2 \text{ Si O}^2 + 3 (\text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2) + 12 \text{ aq} ;]$

Scolezite $[\text{Ca O}, \text{Si O}^2 + \text{Al}^2 \text{ O}^3, \text{Si O}^2 + 9 \text{ aq} ;]$

Prehnite—from Colonel Prehn, its discoverer— $[2 (3 \text{ Ca O}, \text{Si O}^2) + 3 (\text{Al}^2 \text{ O}^3, \text{Si O}^2) + 3 \text{ H O}, \text{Si O}^2 ;]$

Potassa Harmotome $[3 \text{ K O}, 2 \text{ Si O}^2 + 2 (3 \text{ Ca O}, 2 \text{ Si O}^2) + 10 (\text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2) + 45 \text{ aq} ;]$ *et cetera*.

* In Silicates without water ; *exempli gratia*—

Labradorite $[\begin{smallmatrix} 3 \text{ Na O} \\ 3 \text{ Ca O} \end{smallmatrix} \} \text{Si O}^2 + 3 (\text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2) ;]$

Scapolite—*Wernerite*— $[\begin{smallmatrix} 3 \text{ Fe O} \\ 3 \text{ Ca O} \\ 3 \text{ Mg O} \\ 3 \text{ Na O} \end{smallmatrix} \} 2 \text{ Si O}^2 + \{ \begin{smallmatrix} \text{Al}^2 \text{ O}^3 \\ \text{Fe}^2 \text{ O}^3 \end{smallmatrix} \} 2 \text{ Si O}^2 ;]$

Anorthite $[3 \{ \begin{smallmatrix} 3 \text{ Ca O} \\ 3 \text{ Mg O} \end{smallmatrix} \} \text{Si O}^2 + 8 (\text{Al}^2 \text{ O}^3, \text{Si O}^2) ;]$

† In Silicates of Lime and Magnesia, in which the alkaline earths are more or less replaced by Protoxides of Manganese and Iron, and the Silicic acid, sometimes by Alumina. To these belong :—

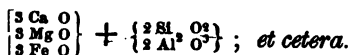
* *Amphibole*—*Hornblende*—according to BONSODORFF, contains hydrofluoric acid ; with Lime, it forms fluoride of Calcium. BERZELIUS gives it the following general formula $[\text{Ca Fl} + 5 (\text{Ca O}, \text{Si O}^2 + 3 \text{ Mg O}, 2 \text{ Si O}^2) ;]$ *Tremolite* or *Grammatite*, *Asbestos Tremolite*, *Asbestos*, *Asbestiform Actynolite*, *Byssolite*, the *Hornblendes*, *et cetera*. Further :—

† Pyroxenes ; *exempli gratia*, *Diopside* or *Alalite*, white *Malacolite*, *Sahlite*, are represented by the formula $[3 \text{ Ca O}, \text{Si O}^2 + 3 \text{ Mg O}, 2 \text{ Si O}^2 ;]$ also,

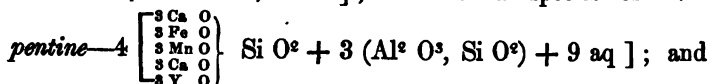
‡ The Ferriferous Pyroxenes, as *Ferriferous Malacolite* $[9 \text{ Ca O}, 6 \text{ Si O}^2 + \{ \begin{smallmatrix} 3 \text{ Fe O} \\ 3 \text{ Mg O} \end{smallmatrix} \} + 2 \text{ Si O}^2 ;]$ the *Hedenbergite* $[\begin{smallmatrix} 3 \text{ Ca O} \\ 3 \text{ Fe O} \end{smallmatrix} \} + 3 \text{ Si O}^2 ;]$ and the *Lieserite*—*Yenite*— $[3 \text{ Ca O}, \text{Si O}^2 + 4 (3 \text{ Fe O}, \text{Si O}^2) ;]$

§ In Silicates of Lime and Magnesia, with Alumina, in which the Alumina is often replaced by Iron and Manganese, *exempli gratia*, in *Zoisite* $[2 (3 \text{ Ca O}, \text{Si O}^2 + 5 \{ \begin{smallmatrix} \text{Al}^2 \text{ O}^3 \\ \text{Fe}^2 \text{ O}^3 \end{smallmatrix} \} \text{Si O}^2) ;]$ *Pistacite*—*Epidote*— $[3 \text{ Ca O}, \text{Si O}^2 + 4 \{ \begin{smallmatrix} \text{Al}^2 \text{ O}^3 \\ \text{Fe}^2 \text{ O}^3 \end{smallmatrix} \} \text{Si O}^2 ;]$ *Idiocrase*—*Vesuvian*—*Egeran*, and many Garnets, the general

formula for which is $[\begin{smallmatrix} 3 \text{ Mg O} \\ 3 \text{ Fe O} \\ 3 \text{ Ca O} \\ 3 \text{ Mn O} \end{smallmatrix} \} \text{Si O}^2 + \text{Al}^2 \text{ O}^3, \text{Si O}^2 ;]$ *Gehlenite*



⁷ In Silicates of Yttria, Oxide of Cerium, Alumina, Lime, Iron, and Manganese, namely, in *Cerine*, which contains Cerium, Silicic acid, Iron, Lime, Alumina, Titanic acid, Manganese, and Water; *Allanite* $[3 \text{ Fe O}, \text{Si O}^2 + 3 \text{ Ce O}, \text{Si O}^2] + 3 \text{ Ca O}, \text{Si O}^2 + 2 \text{ Al}^2 \text{ O}^3, \text{Si O}^2]$; *Orthite*—a species of *Serpentine*—



Pyrorthite $[3 \text{ Ce O}, \text{Si O}^2, + 3 (\text{Al}^2 \text{ O}^3, \text{Si O}^2) + \text{Carbon, Lime, Yttria, Manganese, Iron, et cetera.}$

⁸ In polybasic Silicates, with one or more Borates mixed with them, *exempli gratia*, *Axinite* $[2 \left\{ \begin{smallmatrix} 3 \text{ Ca} & \text{O} \\ 3 \text{ Mg} & \text{O} \end{smallmatrix} \right\}, \text{Si O}^2 + 2 \left\{ \begin{smallmatrix} \text{Al}^2 & \text{O}^3 \\ \text{Fe}^2 & \text{O}^3 \\ \text{Mn}^2 & \text{O}^3 \end{smallmatrix} \right\} \text{Si O}^2] + \left\{ \begin{smallmatrix} \text{B} & \text{O}^3 \\ \text{Si} & \text{O}^3 \end{smallmatrix} \right\}]$; and lastly,—

⁹ In Silicates of Soda, Lime, and Alumina, mixed with Sulphates; *exempli gratia*,—*Lapis Lazuli*, known as *Lazurstein*, *Lazulite*, and *Azure Stone*.

KANE, in treating of this mineral, gives its composition as follows :—

Silica	35·8
Alumina	34·8
Soda	23·2
Sulphur	3·1
Carbonate of Lime	3·1

100·0

and remarks, that it is difficult to deduce a formula from these numerical results, as the state of combination of the sulphur is not well understood. In *Nosin*—*Spinellane*— $\text{Na O}, \text{Si O}^2 + 3 (\text{Li O}, \text{Si O}^2) + 6 (\text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2)]$, *et cetera*. This earth also presents itself in numerous silicates with Soda, Manganese, Potassa, Alumina, Magnesia, Iron, as in *Babingtonite*, *Barsowite*, *Epidote*, *Anorthite*, *Beaumontite*, *Okenite*, *Glottalite*, *Kirwanite*, *Edingtonite*, *Holmite*, *Huronite*.

Leading gangues of ores very often eviscerate Calcareous and Fluor Spars; lime more frequently forms a principal component

of several ores, which are assayed in the dry way, and also an ingredient of the slags, scorise, and recements obtained therefrom.

Examination for Lime.

Fluor spar is very readily recognized before the Blowpipe. When slightly heated, it assumes a luminous appearance, which appears greenish in the dark. On the temperature increasing, it decrepitates. Heated on charcoal as an impalpable powder, it melts to an opaque glass.

Fluor spar can be recognized with the greatest degree of certainty, by heating it with Gypsum, Heavy Spar, and Celestine, whereby it melts in the flame to a clear bead, which is opalescent when cold.

If Calcareous Spar—carbonate of lime—be heated in the Blowpipe flame upon charcoal, carbonic acid escapes, and during its evolution the lime acquires a peculiar lustre. If the residue, which is caustic lime, is treated with water, a great heat is produced, and the mass reacts alkaline. In this combination the temperature rises sufficiently high to inflame gunpowder. When strongly heated in the apex of the blue flame, upon the pincers, the Calcareous Spar, after being causticized, colors the outer flame carmine-red, but much weaker than a strontia salt. This mineral behaves towards borax and microcosmic salt, in which fluxes it is dissolved with effervescence, as also towards soda, like lime. The other varieties of carbonate of lime comport themselves similarly ; but if they contain metallic oxides, they impart to the fluxes the characteristic colors of these oxides.

Arragonite, when smelted on charcoal, decomposes, and fusible particles remain. This residue reacts alkaline on moistened reddened litmus paper, and when held in the apex of the blue flame, between the platinum forceps, the outer flame is colored carmine-red, from the presence of Strontia. Arragonite dissolves, with effervescence, in borax and microcosmic salt, forming a clear glass. It is not at all affected by soda ; the alkali is absorbed by

the charcoal, and the lime assay remains on the surface. When heated to incipient redness, it tumefies, forms a light white powder, and a *very small* quantity of water collects in the beak of the matrass.

Dolomite, Rautenspar, and Brownspar, are infusible before the Blowpipe ; when freed from their carbonic acid they are alkaline. They dissolve in borax and microcosmic salt to a clear glass, generally colored by oxide of iron and manganese, and are not at all affected by soda.

Lime and Magnesia are only recognized with certainty, before the Blowpipe, by the assistance of the moist way, as will be described under Magnesia.

In Baryto-calcite the lime is determined as mentioned under Baryta.

Sulphuric Acid combinations :—

a. Gypsum and Anhydrite are difficultly smeltable in the oxidating flame, to an enamelled glass. If the fusion is performed on charcoal in the reducing flame, they are reduced to sulphides, which, when moistened, have an hepatic smell, and react alkaline with reddened litmus paper. With borax they dissolve with effervescence to a clear glass, which, on cooling, is yellow or dark yellow. When more Gypsum or Anhydrite is added, the bead is brown and opaque when cold. They behave with microcosmic salt and carbonate of soda like pure lime. Gypsum and Anhydrite melt readily to a clear glass, which is enamel-white on cooling ; when the blowing is continued, the bead tumefies and becomes infusible. The characteristics for distinguishing Gypsum from Heavy Spar and Celestine, are as follows :—1. The slight hardness of the Gypsum ; 2. Its ready fusibility, in comparison with the Sulphate of Baryta ; and, 3. Principally, the comportment of Gypsum with carbonate of soda, as regards the baryta and strontia, which are absorbed by the charcoal, while the lime remains.

b. Polyhallite, when heated in a glass tube, liberates water ; on charcoal it fuses to a clouded red globule, which in the reducing flame becomes white, and appears vesicular, its taste is saline and somewhat hepatic. Smelted in the ring of the

platinum wire, it colors the outer flame yellow, from the presence of soda.

With borax it dissolves easily, with violent effervescence, to a transparent glass; but if a large quantity of the mineral has been added, the bead will be colored by the iron, opaque on cooling. It is decomposed by soda, and produces an argillaceous mass, which in the reducing flame assumes a yellowish hue, from being contaminated with hepar—sulphide of antimony.—With fluor spar it melts to an unclear pearl.

Lime and magnesia are not readily separated by oxalate of ammonia and microcosmic salt, from an aqueous solution of the mineral, treated with hydrochloric acid. The contents of potassa can only be found in this case according to the method given under the head of that alkali.

c. Glauberite, when treated *per se* on charcoal, becomes at first white, and then melts to a transparent pearl. This also occurs in the reduction flame, and the assay becomes hepatic. By continuing the blast, the sulphide of sodium is absorbed by the charcoal, and the lime remains.

When the powdered mineral is kneaded into a paste with water, and held on the ring of the platinum wire, in the oxidating flame, the outer sphere is colored yellow, owing to the presence of soda.

When smelted with carbonate of soda on charcoal, the assay is decomposed, and the alkali, with the reduced sulphide of sodium, is absorbed by the support, and the lime remains behind.

d. The pyrognostic properties of Calcareous Heavy Spar, have already been treated of under Baryta, see page 109.

Apatite alone fuses with difficulty on the edges. It dissolves tardily in soda, forming a clear glass; which often partakes of the iron color; when there is a slight excess of the mineral, the assay becomes turbidly streaked, and with a still greater addition, is opalescent when cold.

It dissolves in large quantities in microcosmic salt to a clear glass, which is, however, slightly tinged from the presence of oxide of iron. When slightly saturated, the bead becomes

opaque on cooling, and exhibits facets, which are, however, less perceptible than those produced by phosphate of lead, when heated in the reduction flame and allowed to refrigerate. If the glass be perfectly saturated, a milk-white globule results, and no facets are observable.

With an equal part of carbonate of soda, Apatite produces a vesicular infusible mass, and when more of the alkali is added, it is absorbed by the charcoal. Chlorine, phosphoric, and hydrofluoric acids are determined according to the method given under their separate heads.

The presence of lime can only be ascertained with the assistance of the moist way. The substance should be dissolved in hydrochloric acid, the solution diluted with spirit of wine of about 63°, and the lime precipitated by weak sulphuric acid. The supernatant liquor can, after filtration, be examined with ammonia for alumina. As the compound of lime with phosphoric acid is precipitated by ammonia unchanged, from a solution of the phosphate of lime diluted with water; consequently, alumina and sesquioxide of iron cannot first be precipitated by ammonia. It is necessary to separate the lime first, by means of dilute sulphuric acid.

Pharmacolite is readily discovered by triturating with carbonate of soda, and treating the mixture on charcoal in the reducing flame. The arsenic acid is reduced and volatilizes, which is ascertained by the smell. The soda is absorbed by the charcoal, and the lime remains behind.

Schwerstein—Tungstate of Lime—fuses on the edges to a partially translucent bead.

It dissolves readily in borax to a transparent glass, in the oxidating flame, which soon appears crystalline and milk-white. This assay is not colored when mixed with tin and treated in the reducing flame.

It soon liquefies with microcosmic salt in the oxidating flame, forming a colorless glass, which in the reducing flame partakes, while hot, of a green color, and on refrigeration of a beautiful blue. The glass, when treated with tin on charcoal in the reducing flame, assumes a darker color, which on cooling is green.

By continued treatment in the reducing flame, with a sufficient addition of tin, the tungsten separates, and the glass only exhibits a slight greenish hue.

With carbonate of soda it tumefies, and becomes rounded on the edges. If a small quantity of the pulverized mineral be smelted with four times its volume of soda, on a platinum wire, or in a platinum spoon, and the assay affused over the flame of a lamp with water, tungstate of soda, and the excess of alkali added, will be dissolved, and the lime collects at the bottom of the spoon. The tungstic acid is separated from this solution, according to the method given under the head of this acid, and the lime is determined with soda on charcoal.

Sphene and Titanite, alone before the Blowpipe, slightly intumesce, and fuse on the edges to a dark glass.

With borax they readily melt to a yellowish diaphanous glass, but in the reducing flame the color of titanium is not produced.

In microcosmic salt, Sphene and Titanite dissolve with difficulty; the undissolved part becomes opaline. The amethystine tinge is afforded, particularly when the assay is treated upon charcoal mixed with tin in the reducing flame. With carbonate of soda they fuse to an opaque glass, but in no proportion give a transparent one; on cooling, the globule is white, or greyish white. When an excess of the alkali has been added, the greater part of the assay is absorbed by the charcoal, and the lime remains behind.

If Sphene or Titanite be smelted with six times its volume of bisulphate of potassa, in a platinum spoon, and the melted mass affused over the flame of a lamp with a sufficient quantity of water,—the liquid must not be allowed to boil,—the titanic acid and sulphate of lime dissolve, leaving a silicious residue. The titanic acid deposits from the filtered solution upon boiling, and the lime can be separated by oxalate of ammonia. Should there be any doubt as to the nature of the precipitates, they can be separately examined, after being well washed, before the Blowpipe,—that is, the lime with carbonate of soda on charcoal, and the titanic acid according to the method given under the head of Titanium.

The silicious deposit may contain titanitic acid, and a portion of sulphate of lime, if the mineral had not been previously reduced to a fine powder, or if an adequate quantity of water was not added.

The pyrognostic properties of Yttrite-tantalite are given under Yttria.

Datholite and Botryolite—both from Arendal, in Norway—behave before the Blowpipe, according to BERZELIUS, as follows:—

When treated alone in tubes, they evolve aqueous vapor. On charcoal they vesiculate like borax, and then melt into a transparent globule, which sometimes, from impurity or unequal limpidity, is either rose-red or iron-green. They dissolve readily in borax to a clear bead, without assuming the colors above mentioned.

With microcosmic salt they easily fuse, leaving behind a silicious compage; a greater addition of the assay gives at first an opaque glass, and lastly, an enamel-white one.

They dissolve with a little soda to a clear glass; on a greater addition the bead is opaque on cooling, and with an excess the whole assay is absorbed by the charcoal.

With gypsum, they fuse with greater difficulty than Fluor Spar, to a clear globule, which remains so on cooling.

With a solution of nitrate of cobalt, Datholite and Botryolite give an unclear blue glass. The lime is determined in these minerals by smelting with bisulphate of potassa, and treating the glacial residue with an adequate quantity of water, and decomposing the clear solution, after separation of the silica with oxalate of ammonia. The boracic acid is recognized by a special test.

In Silicates, the aid of the moist way is always requisite for determining the lime, which is very seldom ascertained with certainty before the Blowpipe.

This earth, which forms the only base of Tabular Spar, can be readily found by melting the powdered mineral with an excess of bisulphate of potassa, and treating the fused mass with sufficient water. The sulphate of lime formed, and the sulphate of potassa,

dissolve, while the silica remains behind, sometimes with a portion of the gypsum. The earth is precipitated after filtration by oxalate of ammonia; the oxalate of lime is then collected on a filter, and, after being washed, treated with soda on charcoal, or with microcosmic salt upon the platinum wire. Should the mineral contain traces of alumina, this earth can be precipitated by caustic ammonia, before the lime is separated by the oxalate, and afterwards tested with a solution of cobalt, provided there is no sesquioxide of iron present to prevent the pale-blue reaction, upon charcoal. To obtain the silica quite free from sulphate of lime, the silicious residue on the filter must be treated with a *very weak* hydrochloric acid solution, and filtered. This second filtrate must not be added to the first, because if it is not properly diluted, a precipitate of sulphate of lime may occur on the addition of ammonia. Silicates,—including furnaced ores, scorise, *et cetera*, with several bases, are sometimes not at all decomposed by bisulphate of potassa. This, however, is effected, if the process be conducted similar to that given under Baryta for furnaced ores and slags.—For the decomposition of silicates, a mixture of carbonate of soda and potassa will be found the best.

If the solution obtained after melting the silicate with soda and borax, *et cetera*,—given under Baryta, contains protoxide of iron, it must be converted into the sesquioxide with a few drops of nitric acid; then add chloride of ammonium, and precipitate the alumina and sesquioxide of iron by a slight excess of ammonia. The method of separating these two constituents will be given under Alumina, when the silicious compounds are noticed. The lime which is in solution, combined probably with portions of magnesia, protoxide of manganese, and oxide of cobalt, is examined as follows. Sulphide of Ammonium is added to the solution, until all the manganese and cobalt are precipitated. The liquid is then filtered, and the residue washed with water, containing some of the precipitant. After affusing for some time, the filter containing the sulphides is burnt, the residue pulverized, and examined with microcosmic salt on platinum wire for cobalt, and with carbonate of soda, to which a small quantity of saltpetre

has been added, on the platinum foil, in the oxidating flame, for manganese.

To the filtrate is added a few drops of hydrochloric acid, and the whole is treated over the lamp, until no sulphide of hydrogen escapes. The menstruum is then filtered from the sulphur, the solution saturated with ammonia, the lime precipitated as an oxalate, and separated by filtration. The magnesia is precipitated by microcosmic salt as ammonia-phosphate, but before adding the microcosmic salt it must be ascertained whether all the lime has been abstracted. When no manganese or cobalt is present, there will only be two filtrations, to separate the lime from the magnesia. If the silicate contains traces of protoxide of manganese only, the precipitated magnesia must be examined for them with soda and saltpetre, as will be given under Manganese.

The protoxide of manganese is converted into a higher oxide by the nitre which combines with the alkali, forming a clear limpid glass, which is driven from one part of the platinum foil to another, while the magnesia remains in one spot upon the support, and is readily seen.

The examination of silicates, which, besides lime, may contain yttria and protoxide of cerium, is given under Yttria.—When solutions of lime are so very dilute, that sulphuric acid causes no precipitate, alcohol should be added, when a deposit of sulphate immediately appears.

DOBEREINER has given an excellent method for the separation of lime and magnesia, which is nearly as follows :—Evaporate the hydrochloric acid solution of the two earths to dryness, in a large porcelain crucible, and then ignite to expel the excess of acid. Add chlorate of potassa in small crystals, until the eliminated chlorine gas is perceived. When the dry mass is affused with water, chlorides of calcium and potassium dissolve, and a residue of pure hydrated magnesia remains. The liquid can now be filtered, to separate the magnesia, and oxalate of ammonia added, to precipitate the lime.

§ 8. *MAGNESIA*—Mg O—*Presence in the Mineral Kingdom.*

This earth occurs as follows :—

a. Per se, as Periklas.

b. In Aluminate, as also in Aluminate of Magnesia—Spinel—[Mg O, Al² O³]; and as an Aluminate of the Protoxide of Iron and Magnesia, in *Zeylanite—Pleonaste*— $\left[\begin{smallmatrix} \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \end{smallmatrix} \right\} \text{Al}^2 \text{O}^3$];

c. In Brucite—Condrodite, Maclureite—[Mg Fl + 3 (3 Mg O, Si O²)] ;

d. With Carbonic Acid, in Magnesite $\left[\begin{smallmatrix} \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \\ \text{Mn} & \text{O} \end{smallmatrix} \right\} \text{C O}^2$]; and with Carbonic acid and Carbonate of Lime, in many *Carboniferous Spars*, as Dimeric in *Dolomite*, Isomeric in *Tarandite*, Americ in *Brown Spar, et cetera.*

e. With Sulphuric Acid and Water, in Sulphate of Magnesia [Mg O, S O³ + H O + 6 aq] ; and with Sulphuric Acid, Sulphate of Potassa, and Sulphate of Lime, in *Polyhallite.*

f. With Boracic Acid, in Boracite [Mg O, 2 B O³, + 2 (Mg O, B O³)] ;

g. In Silicates :—

¹ In which Magnesia forms the principal base ; *exempli gratia*, in *Speckstein* [6 Mg O, 5 Si O² + 4 aq] ; *Meerschäum* [Mg O, Si O² + aq] ; *Picrosmine—Parallolite*—[3 Mg O, 2 Si O²] ; *Serpentine*—hydrate of magnesia with subsilicate of magnesia—[3 Mg O, 6 H O + 2 (3 Mg O + 2 Si O²)] ; *Olivine* [3 Ca O, Si O² + $\left[\begin{smallmatrix} 3 & \text{Mg} & \text{O} \\ & \text{Fe} & \text{O} \end{smallmatrix} \right\} \text{Si O}^2$,] *et cetera.*

² In Silicates of Lime and Magnesia, with Silicates of Alumina, without water ; as *Anorthite* [3 Mg O, Si O² + 2 (3 Ca O, Si O²) + 8 Al² O³, Si O²] or [3 Mg O, Si O² + 2 Al² O³, Si O² + 2 (3 Ca O, Si O² + 3 Al² O³, Si O²)]—*BERZELIUS*, and according to *ABICH* [3 R O, 2 Si O² + 2 (R² O³, 2 S O²) + 6 (3 Ca O, Si O²) + 3 (Al² O³, Si O²)] ;

³ In Silicates of Potassa or Soda, with Silicates of Magnesia, Alumina, and Protoxide of Iron, with and without water, in which the alkalies are often replaced by Magnesia and Protoxide of

Iron ; *exempli gratia*, in *Prismatic Magnesian Mica*, to which *Potstone*, *Green Earth*, *Chlorite*, and *Talc* belong ;

⁴ In Silicates of Lime and Magnesia, in which the alkaline earths are more or less replaced by Protoxides of Iron and Manganese, and the Silicic acid sometimes by Alumina ; to these belong *Amphibole* and the *Magnesia-Pyroxenes* ;

⁵ In Silicates of Lime and Magnesia with Alumina, in which the alkaline earths are sometimes replaced by Protoxide of Iron, and the Alumina by the Sesquioxide ; *exempli gratia*, in *Magnesia-Idiocrase* ; in the *Black Garnet of Arendal* ; in *Soapstone* [$3 \text{ Mg O}, 2 \text{ Si O}^2 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2 + 6 \text{ aq}$] ; in *Blue* and *Red Dichroite*—*Iolite*, *Cordierite*— $\left[\begin{smallmatrix} 3 \text{ Mg O} \\ 3 \text{ Fe O} \\ 3 \text{ Mn O} \end{smallmatrix} \right] \text{ Si O}^2 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2$ and [$3 \text{ Mg O}, 2 \text{ Si O}^2 + \left\{ \begin{smallmatrix} 3 \text{ Al}^2 \text{ O}^3 \\ 3 \text{ Fe O} \\ 3 \text{ Mn O} \end{smallmatrix} \right\} \text{ Si O}^2$] ;

⁶ In Silicates with Phosphates ; *exempli gratia*, in *Sordawalite*, seemingly a mixture of [$2 \text{ Mg O}, \text{P O}^2$] with [$3 \text{ Mg O}, 2 \text{ Si O}^2 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2 + 2 (3 \text{ Fe O}, 2 \text{ Si O}^2 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^2)$] ;

⁷ In Silicates with metallic Fluorides ; as in *Chondrodite* [$3 (\text{Mg Fl}) + 4 (3 \text{ Mg O}, \text{Si O}^2)$].—Magnesia is met with in *Boltonite*, *Batrachite*, *Nemalite*, *Villarsite*, *Steatite*, *Aphrodite*, *Gymnite*, *Antigonite*, *Pennine*, *Monradite*, *Piotine*, *Leuchtenbergerite*, *Spadaite*, *Schillerspar*, *Dermatine*, *Praseolite*, *Esmarkite*.

If some of the above mentioned minerals occur as well in the gangues as in the ores, the ores prepared in the dry way, and also the recrements and scoræ obtained therefrom, generally contain a small quantity of Magnesia, which can readily be determined.

Examination for Magnesia.

a. Aluminate, Spinel, and Zeylanite are readily recognized before the Blowpipe, as they give characteristic glasses, owing to the metallic oxides they contain, but magnesia *per se*, is not detectable with certainty. To arrive at this earth, the finely powdered mineral must be heated with two parts of soda and three parts of borax, upon charcoal, the fused mass dissolved in

hydrochloric acid, the solution evaporated to dryness; again dissolved in water, and the liquid filtered, to separate the silica. Nitric acid must then be added to the filtrate, to convert the protoxide of iron into sesquioxide, and two spoonfuls of chloride of ammonium introduced; the iron, alumina, and any oxide of chromium must then be precipitated by ammonia, and separated from the supernatant liquor. The filtrate must now be boiled, the magnesia precipitated by microcosmic salt as ammonia-phosphate. This precipitate must be collected on a filter, edulcorated with water, and then treated on charcoal. It fuses to a white crystalline translucent pearl, and when treated with a few drops of cobalt solution, assumes a violet color, which appears reddish in the flame of the lamp. When the Spinel dissolves in borax with effervescence, it is a sign that there is some carbonate of lime between the interstices. Should this occur, the lime is to be precipitated by oxalate of ammonia, before the addition of the microcosmic salt. The aluminous precipitate can be examined with borax or microcosmic salt upon the platinum wire, for iron and chromium, or else treated with potassa, which dissolves the alumina, and leaves the sesquioxide of iron as a reddish brown, and the oxide of chromium as a dirty green deposit. Another method of analysing a mixture containing alumina, sesquioxide of iron, and oxide of chromium, is as follows:—To a cold hydrochloric acid solution of the compound, add solution of potassa, which will precipitate the sesquioxide of iron as a reddish brown powder, and filter. Boil the solution, which should contain an excess of alkali; the chromium will precipitate of a green color, and must be separated by filtration. To the filtrate add chloride of ammonium, which will throw down the alumina. This is an excellent method, especially for qualitative analysis.

If the mineral contains iron and chromium, and the residue only appears ferruginous, the mass may be smelted upon the platinum wire with nitrate of potassa, and the fused mass treated with water. The chromate of potassa formed, and the undecomposed nitrate, dissolve, while the iron remains behind. The chromic acid can be detected in the solution, according to the method given under Chromium.

b. Brucite, when moistened, after being treated on charcoal, reacts alkaline with reddened litmus paper. It comports itself towards fluxes similarly to magnesia.

c. Carbonate of Magnesia :—

¹ Magnesite, when not contaminated, behaves like Brucite :

² Carbonate of Magnesia with Carbonate of lime, as Dolomite, Brown Spar, *et cetera*.

These compounds behave like carbonate of lime before the Blowpipe, so that the magnesia cannot be distinguished from the lime. It must, therefore, be obtained after separating the iron and lime, by the process previously given as ammonia-phosphate, and then treated on charcoal *per se*, and also with equal parts of soda and nitre upon the platinum wire. It ought to melt to a white crystalline pearl, easily soluble in microcosmic salt, provided no trace of manganese is present, producing a colorless glass, remaining so when brought in contact with a crystal of saltpetre. The smallest portion of manganese, if present, will be infallibly detected by treating the precipitated magnesia with soda and nitre on platinum foil.

If an appreciable quantity of manganese is contained in the specimen, it is better to precipitate it by sulphide of ammonium ; then filter, and free the solution from sulphide of hydrogen, as given in the lime examination.

d. Sulphate of Magnesia—Epsomite—is immediately recognized on treating with a solution of nitrate of cobalt.

e. Boracite fuses with intumescence on charcoal ; it is difficult to obtain a clear pearl, as it becomes drusy upon cooling ; that is, the whole surface of the charcoal presents crystalline needles. The glass while hot appears slightly yellowish, from the presence of sesquioxide of iron, but on refrigeration it is opaline. It readily dissolves in borax, forming a transparent ferruginous colored glass.

Boracite also dissolves with facility in microcosmic salt to a clear glass, here and there turbidly streaked, and if an excess is present, the bead will be opaque when cold.

When it is melted with carbonate of soda sufficient to produce a clear glass, the assay on cooling presents crystals and large

facets. With more of the alkali, the glass is similar to a magnesian bead not sufficiently saturated. Heated in a powdered state upon charcoal, with a few drops of a cobalt solution, the whole concretes forming a blue mass.

When melted with bisulphate of potassa, and the fused assay dissolved in water, the magnesia can be precipitated and examined as before stated.

Should the Boracite contain borate of lime also, it is readily discovered by adding to the aqueous sulphate solution a little chloride of ammonium, and then oxalate of ammonia, before precipitating the magnesia with microcosmic salt.

The Boracic Acid is determined according to the process given under this head.

f. In Silicates, in which magnesia forms the only base, *exempli gratia*, Speckstein, Meerschaum, Picrosmine, and Serpentine, the magnesia is ascertained the most readily by smelting the mineral with bisulphate of potassa, and treating the vitreous residue with water; by this means the insoluble silicic acid is separated, and the filtrate, containing sulphate of magnesia, can be heated with microcosmic salt, and examined as before remarked. If the mineral contains lime, as Olivine, it must be separated previously to precipitating the magnesia. Silicates containing several bases, to which the majority of the prepared ores and slags belong, cannot have the magnesia determined with certainty before the Blowpipe, either alone or with bisulphate of potassa; therefore, they must be examined in the same manner as given under Baryta and Lime. The precipitation of the Basic Ammonia-phosphate of Magnesia, is greatly facilitated when the menstruum is boiled. When there is a *very small* quantity of magnesia present, it is better to allow the liquid, after the addition of the precipitant, to repose for some hours.

§ 9. ALUMINA— $Al^3 O^3$ —*Presence in the Mineral Kingdom.*

Alumina is very abundant, being found,—

a. As pure Alumina, with slight traces of Silica and Ses-

quioxide of Iron, in *Corundum*, *Sapphire*, *Ruby*, and *Diamant Spar* ;

b. As Hydrate of Alumina, in *Diaspore* [$\text{Al}^2 \text{O}^3 + \text{aq}$] ; and *Gibbsite* [$\text{Al}^2 \text{O}^3 + 3 \text{aq}$] ;

c. With Magnesia, as Magnesia-Alumina, in *Spinel* ; with Protoxide of Iron, as Ferruginous Aluminate of Magnesia, in *Zeylanite* ; with Oxide of Zinc, *et cetera*, Zincous Alumina, in *Automalite*—*Gahnite*— $\left[\begin{smallmatrix} \text{Zn} & \text{O} \\ \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \end{smallmatrix} \right] \text{Al}^2 \text{O}^3$; and with Oxide of Lead,

as Aluminate of Lead, in *Plombgomme* [$\text{Pb O}, \text{Al}^2 \text{O}^3 + 6 \text{aq}$] ;

d. With Fluorine and Fluoride of Sodium, in *Cryolite* [$3 (\text{Na Fl}) + \text{Al}^2 \text{O}^3, 3 \text{Fl}$] ;

e. With Sulphuric Acid and Water, in *Aluminite* [$\text{Al}^2 \text{O}^3, \text{S O}^3 + 9 \text{aq}$] ; with Sulphuric Acid, Potassa, and Water, in *Potassa-Alum* ; with the same constituents without Potassa, but with Ammonia, in *Ammonia-Alum* ; and with Sulphuric Acid, Potassa, Sesquioxide of Iron, and Water, in *Iron-Alum* ;

Formulæ for the two above-mentioned Alums :

Potassa-Alum [$\text{K O}, \text{S O}^3 + \text{Al}^2 \text{O}^3, 3 \text{S O}^3 + 24 \text{aq}$] ;

Ammonia-Alum [$\text{N H}^4 \text{O}, \text{S O}^3 + \text{Al}^2 \text{O}^3, 3 \text{S O}^3 + 24 \text{aq}$] ;

Iron-Alum [$\text{K O}, \text{S O}^3 + \text{Fe}^2 \text{O}^3, 3 \text{S O}^3 + 24 \text{aq}$] ;

f. With Phosphoric Acid and Lithia, in *Amblygonite* : with Phosphoric Acid, Water, a little Fluorine, Lime, Protoxides of Iron and Manganese, in *Wavellite* [$2 \text{Al}, 3 \text{Fl} + 3 (4 \text{Al}^2 \text{O}^3, 3 \text{P O}^5) + 18 \text{aq}$] ; with Phosphoric Acid, Magnesia, Silica, Protoxide of Iron, and Water, in *Lazulite* [$3 (5 \text{Mg O}, 2 \text{P O}^5) + 4 (5 \text{Al}^2 \text{O}^3, 3 \text{P O}^5) + 4 \text{Fe O}, \text{P O}^5 + 15 \text{aq}$] *et cetera*, in varying proportions ;

g. In Silicates, *exempli gratia*.

¹ Where Alumina is the only base, as in *Cyanite* [$2 \text{Al}^2 \text{O}^3, \text{Si O}^2, + 3 (\text{Al}^2 \text{O}^3, \text{Si O}^2)$] ;

² In Silicates of Potassa and Lime ; *exempli gratia*, in *Zoisite*—*Epidote* ;

³ In Silicates of an Alkali, or of an Alkaline Earth, and Silicates of Alumina, combined with water of crystallization. To these belong *Chabasite* $\left[\begin{smallmatrix} 3 \text{Ca O} \\ 3 \text{Na O} \\ 3 \text{K O} \end{smallmatrix} \right] \text{Si O}^2 + 3 (\text{Al}^2 \text{O}^3, 2 \text{Si O}^2) +$

15 aq] ; *Mesolite* $[\text{Na O, Si O}^2 + \text{Al}^2 \text{O}^3, \text{Si O}^2 + 2 \text{ aq} + 2 (\text{Ca O, Si O}^2 + \text{Al}^2 \text{O}^3, \text{Si O}^2 + 6 \text{ aq})]$,—*Mesolite* may be considered as a mixture of *Mesotype* and *Scolezite* ; *Mesotype* $[\text{Ca O, Si O}^2 + \text{Al}^2 \text{O}^3, \text{Si O}^2 + 3 \text{ aq}]$; *Anaclime* $[3 \text{ Na O, } 2 \text{ Si O}^2 + 3 (\text{Al}^2 \text{O}^3, 2 \text{ Si O}^2) + 6 \text{ aq}]$; *Stilbite* *Lamonite*, *Harmotome*, *Prehnite*, *et cetera*.

⁴ In Silicates of the Alkalies or Alkaline Earths, with Silicates of Alumina, without water ; *exempli gratia*, *Felspar*, *Albite*, *Petalite*, *Spodumene*, *Leucite*, *Labradorite*, *Scapolite* ; the formula of the last is $[\frac{3}{3} \frac{\text{Ca}}{\text{Na}} \frac{\text{O}}{\text{O}}] 2 \text{ Si O}^2 + 2 (\text{Al}^2 \text{O}^3, \text{Si O}^2)$; further, in *Elaolite* $[\frac{3}{3} \frac{\text{K}}{\text{Na}} \frac{\text{O}}{\text{O}}] \text{ Si O}^2 + 3 (\text{Al}^2 \text{O}^3, \text{Si O}^2)$; *Sodalite*, *Anorthite*, *et cetera* ;

⁵ In Silicates of Potassa or Lithia, with Silicates of Magnesia, Alumina Protoxide of Iron, and Manganese, without water ; in *Mica*, which sometimes contains Fluorine ;

⁶ In Silicates of Potassa or Soda, with Silicates of Magnesia, Alumina, and Protoxide of Iron, with and without water, in which the alkalies are more or less replaced by magnesia or protoxide of iron ; *exempli gratia*, in *Potstone*, *Green-earth*, *Chlorite*, and *Talc* ;

⁷ In Silicates of Lime and Magnesia with Alumina, in which the alkaline earths are often replaced by the protoxides of iron and manganese ; as in *Pistacit*, *Idiocrase* ; in many varieties of *Garnets* ; in *Gehlenite*, *Soapstone* *Dichroite*, and *Karpholite* ; the last sometimes contains traces of *Fluorine*, its general formula is $[\frac{3}{3} \frac{\text{Mn}}{\text{Fe}} \frac{\text{O}}{\text{O}}] \text{ Si O}^2 + 3 (\text{Al}^2 \text{O}^3, \text{Si O}^2) + 6 \text{ aq}$;

⁸ In Silicates of Alumina and Metallic Oxides ; *exempli gratia*, in *Staurolite*, from St. Gothard $[\frac{3}{3} \frac{\text{Al}^2}{\text{Fe}^2} \frac{\text{O}^3}{\text{O}^3}] [\frac{3}{3} \frac{\text{Si}}{\text{Al}^2} \frac{\text{O}^3}{\text{O}^3}]$; and *Allophane*, from Schneeberg, which contains Oxide of Copper.

⁹ In Silicates containing Glucina ; for instance, *Emerald* and *Beryl*, the formula for both of which is $[\frac{3}{3} \frac{\text{G}^2}{\text{Al}^2} \frac{\text{O}^3}{\text{O}^3}] 8 \text{ Si O}^2$. When the former is tinged green by Oxide of Chromium, it is the true *Emerald*, but when perfectly colorless and transparent, it is the *aqua marina*, which is a most valuable gem : further, in *Euclase* $[\frac{\text{Al}^2}{\text{G}^2} \frac{\text{O}^3}{\text{O}^3}] \text{ Si O}^2$; and in *Cymophane*, or *Chrysoberyl* $[4 \text{ Al}^2 \text{O}^3, \text{Si O}^2 + 2 (\text{G}^2 \text{O}^3, 4 \text{ Al}^2 \text{O}^3)]$;

¹⁰ In Silicates which contain *Yttria* and Oxide of Cerium ; as *Cerine*, *Allanite*, *Orthite*, and *Pyrrorthite* ;

¹¹ In Silicates containing Fluorides ; for example, *Topaz* [$\text{Al}^2 \text{O}^3 + 2 (2 \text{Al} 3 \text{Fl}) + 6 (\text{Al}^2 \text{O}^3, \text{Si O}^2)]$;

¹² In Polybasic Silicates, which contain small quantities of one or more Borates, as the various *Tourmalines* [$\text{K O}, \text{Li O}, \text{Na O}, \text{Ca O}, \text{Mg O}, \text{Fe O}, \text{Mn O}, \text{Al}^2 \text{O}^3, \text{Si O}^2, \text{B O}^3$] ; and in *Axinite*. O'SULLIVAN has examined many of the *Tourmalines*, and always found in them more or less Manganese and Lime.

¹³ In Silicates of Soda, Lime, and Magnesia, combined with Sulphates ; as, *Lazulite*, *Nosin*, *et cetera*. Alumina is also met with in *Kryolite*, *Bleigummi*, *Pissophane*, *Thonerde*, *Peganite*, *Striegisane*, *Variscite*, *Honeystone*, *Andalusite*, *Sillimanite*, *Bucholsite*, *Xenolite*, *Ramlite*, *Worthite*, *Kollyrite*, *Miloschine*, *Phole-rite*, *Tuesite*, *Gilbertite*, *Anauxite*, *Kaolin*, *Halloysite*, *Nakrite*, *Ottrelite*, *Cimolite*, *Bolus*, *Plinthite*, *Orthite*, *Malthacite*, and *Pyknite*.

h. In various kinds of rocks ; *exempli gratia*, *Fuller's Earth*, *Common Clay*, *Loam*, *Marl*, *Porphyry*, *Garnet*, *Mica Slate*, *Clay Slate*, *Alum Slate*, *Sandstone*, *Gneiss*, *et cetera*. Clay Slates, *et cetera*, besides the usual given constituents, contain traces of Sulphur, Phosphoric Acid, Chlorine, and Fluorine.

As metallic gangues are generally classed under one or the other of the above mentioned rocks, and as, in obtaining the ores, the adhering stony matrix cannot be completely separated from the former, therefore, it is that alumina generally forms a considerable constituent of many ores prepared in the dry way, and, consequently, an ingredient of the slags obtained in the smelting of such ores.

Examination for Alumina.

a. Corundum, Sapphire, Ruby, and Diamant Spar comport themselves before the Blowpipe, as follows :—

Alone, they remain perfectly unchanged, as well in a powdered state as in fragments.

With borax they fuse perfectly, though with difficulty, to

transparent colorless glasses, which do not become streaked with dull laminæ.

With microcosmic salt they fuse to a clear glass, only when added to the flux in a powdered state.

They are not attacked by soda.

When moistened with a solution of nitrate of cobalt, and held in the state of a fine powder for a long time in the oxidating flame, they produce a beautiful blue color.

By these reactions before the Blowpipe, alumina may be immediately recognised.

b. Hydrate of Alumina :—

¹ Diaspore, when heated in a matrass, decrepitates violently, and splits into small white brilliant scales, without giving out much water. These scales behave themselves with fluxes like pure alumina. Exposed to heat in the oxidating flame, with a few drops of a cobalt solution, this mineral affords a beautiful blue hue.

² Gibbsite, when heated in a flask, yields much water. It is infusible on charcoal, and behaves with fluxes like pure alumina, affording the special appearance with a solution of cobalt.

c. Aluminate :—

¹ Spinel and Zeylanite. The method for the examination of these minerals for alumina has been already described under Magnesia. The precipitate obtained by means of ammonia, after being well affused with water, should be treated with a solution of potassa, and heated in the same manner as in the examination of silicates for alumina,—which will be hereafter given, the solution diluted with water, filtered from the remaining oxides, decomposed with an adequate quantity of chloride of ammonium, and boiled, by which the alumina is thrown down. This precipitate can be tested with a solution of cobalt, after being purified by water.

² Automalite—Gahnite. This mineral, when heated on charcoal *per se*, is unalterable. With borax and microcosmic salt, even when in the state of an impalpable powder, it is with difficulty fused. The resulting beads present the ferruginous tinge. It is not fusible with soda ; it scintillates and affords a dark slag ;

when this is reduced to a very fine powder, mixed with soda, and placed on charcoal in a good reducing flame, the charcoal becomes coated with a ring of oxide in the commencement of the operation. It gives no indication of manganese on a platinum foil.

With borax and carbonate of soda together, it fuses to a clear glass, which becomes opaque and dark green on cooling.

A perfectly pure fragment of this mineral, from Sweden, was found to consist of alumina, oxide of zinc, magnesia, protoxide of iron, and silicic acid.

The analysis was performed in the following manner :—About 75 milligrammes of the fragment, very finely powdered, were melted into a bead, with five times as much of a mixture consisting of two parts of soda and three of borax, on charcoal in the reducing flame. The bead was kept in the inner flame, until all the oxide of zinc was reduced, and the metal volatilized. The charcoal thus became coated with a sublimate of oxide of zinc, which when moistened with a solution of nitrate of cobalt, and ignited in the oxidating flame, afforded on cooling a green color. The fused bead was removed from the charcoal, placed on another piece of charcoal, and kept in a state of fusion in the reducing flame for some time, in order to be certain that all the oxide of zinc was reduced and volatilized. But as no sublimate of oxide of zinc formed, the melted bead was thrown on the anvil, by inverting the piece of charcoal, in order to obtain it free from carbonaceous particles. On cooling, it afforded a copperas-green color, and remained transparent. It was then pulverized, dissolved in hydrochloric acid, the solution evaporated to dryness, and the dry chloride dissolved in water. The aqueous solution was filtered, when a small trace of silicic acid remained behind on the filter, which gave with carbonate of soda the proper reactions. The solution was then heated with a few drops of nitric acid, in order to convert the protoxide of iron into the sesquioxide, and a little chloride of ammonium dissolved in it to prevent the precipitation of magnesia by ammonia. Ammonia was then dropped into the solution until it was over-saturated ; by which a flocculent precipitate of a yellowish-white color was thrown down, which, on being filtered, well washed, and subse-

quently treated with potassa,—as will be mentioned in the examination of silicates for alumina,—proved to be oxide of iron and alumina. To the filtered liquid oxalate of ammonia was added, which produced no milkiness from the precipitation of oxalate of lime; but an addition of phosphate of soda and ammonia determined a precipitate, which, on being filtered, proved before the Blowpipe to be basic phosphate of ammonia and magnesia.

³ Plombgomme. This mineral gives the following reactions before the Blowpipe, from which its constituents can be readily determined.

Heated alone in a flask, it gives off aqueous vapors, and sometimes decrepitates with violence.

On charcoal it loses its transparency, becomes white, swells up like a Zeolite, and semifuses under a high temperature, without, however, being rendered fluid.

With borax and microcosmic salt, it fuses into a colorless transparent glass. The microcosmic bead becomes, with a certain addition of the assay, opaque on cooling.

It does not fuse with soda; small lead globules, however, appear on all sides when the experiment is made on charcoal. It gives a beautiful pure blue color with a solution of cobalt.

d. Fluoride of Sodium with Fluoride of Aluminum; as Cryolite, from Greenland:—

Heated alone in the matrass, it affords some water, and decrepitates without losing its transparency.

Held with the platinum forceps in the apex of the blue flame, it colors the exterior flame a strong yellow, from soda.

Blown upon in an open tube,—the flame being conducted into the tube,—it strongly attacks the glass, and the moisture which collects reacts like hydrofluoric acid. It readily fuses on charcoal to a clear bead, which becomes turbid on cooling. After long exposure to the flame, the bead spreads, the fluoride of sodium is absorbed by the support, and an aluminous crust remains on the surface.

It readily fuses, even in large quantities, to a clear glass, with borax and microcosmic salt, which on cooling becomes milk-white. According to BERZELIUS, the bead with microcosmic salt, treated

in the reducing flame, should sometimes become somewhat red on cooling, from its containing a trace of copper.

It fuses with carbonate of soda to a clear glass, which expands and becomes milk-white on cooling.

A small quantity of the powdered mineral, moistened with a solution of nitrate of cobalt, fuses in the oxidating flame, on charcoal, to a blue bead.

e. Combinations of Alumina with Sulphuric Acid ; as Potassa and Ammonia-Alum, Aluminite, and Alum-stone :—

If the first two salts be heated in a glass flask, they tumefy and give off water ; by stronger heat the Potassa-Alum gives off sulphurous acid, and sulphate of ammonia sublimes from the Ammonia-Alum, which is for the most part dissolved in water ; sulphurous acid is also given off in this case. The residual dry mass from both salts gives, with a solution of nitrate of cobalt, a bright blue color, and behaves with fluxes like alumina. Aluminite yields at first much water, and as it approaches ignition eliminates sulphurous acid, which may be known by its smell, and by its reaction with moistened litmus paper.

It behaves with fluxes, and with solution of cobalt, exactly as alumina.

Alum-stone at first yields water, and by stronger heat a sublimate of sulphate of ammonia, which is soluble in water. The crystallized specimens decrepitate, affording a fine powder.

On charcoal it contracts when exposed to a strong flame, but does not melt.

With microcosmic salt it is easily melted, leaving behind a skeleton of silicic acid.

f. Combinations of Alumina with Phosphoric Acid :—

In these combinations, to which Wavellite, Lazulite, and Amblygonite belong, alumina is readily distinguished by the behavior of these minerals to fluxes, and particularly to a solution of nitrate of cobalt, with which they produce a beautiful blue. It is required, however, to find the other constituents, namely, the lime in Wavellite, and the magnesia in Lazulite. These minerals, as they are not easily soluble in hydrochloric acid, must be first fused to a glass, with equal parts of borax and soda, on charcoal

in the oxidating flame : this glass should then be pulverized, dissolved in hydrochloric acid, the protoxide of iron converted by means of nitric acid into sesquioxide ; the solution,—as it contains phosphoric acid,—diluted with alcohol, and the lime precipitated by dilute sulphuric acid, the sesquioxide of iron and alumina by ammonia, and the magnesia by phosphate of soda and ammonia. Alumina sometimes falls with the sesquioxide of iron, as phosphate of alumina ; it can, in this case, be separated from sesquioxide of iron by means of potassa, and precipitated from the warm diluted solution by chloride of ammonium, as will be immediately mentioned in the case of silicates and varieties of rocks. The silicic acid in Lazulite is detected by treating with hydrochloric acid the specimen melted with soda and borax ; the phosphoric acid in both minerals, as well as the fluorine, and the protoxide of manganese in Wavellite, are detected by special experiments, which are given in different parts of this book, for these minerals ; and the protoxide of iron in Wavellite and Lazulite may be detected by treating them with borax, and also in the precipitate obtained by ammonia.

g. Silicates and Rocks :—

The alumina contained in silicates, and in the different kinds of rocks to which the picked ores and slags belong, cannot be well detected, except in a few of its combinations, by its behavior to vitreous fluxes and solution of nitrate of cobalt. In most cases it is necessary to employ the method which is given under Baryta and Lime.

The precipitate thus obtained by ammonia, which usually consists of alumina and sesquioxide of iron, and also very slight traces of magnesia, or protoxide of manganese, must, when the substance under investigation contains much of these bodies, be well washed on the filter, and heated with potassa while still moist in a porcelain basin, until the whole of the alumina is dissolved out, and the oxide of iron appears in the solution in a finely divided state, of a reddish-brown color, pure or mixed with slight traces of precipitated magnesia, or protoxide of manganese, which, in a qualitative examination, may be entirely overlooked. The alkaline solution of the alumina is then to be diluted with

water, filtered from the oxide of iron, mixed with an excess of hydrochloric acid, and slightly boiled, in order to reprecipitate the alumina ; or it may be supersaturated with hydrochloric acid, and the alumina precipitated by a solution of carbonate of ammonia. The precipitated alumina, after being filtered and well washed, can be tested with solution of cobalt before the Blowpipe. If the substance contains no iron, the precipitate by ammonia appears perfectly white, and need not in this case be treated with potassa, but tested immediately with solution of cobalt.

Silicates containing glucina, yttria, and oxide of cerium, in which it is intended to seek for these substances at the same time, should be treated according to the methods which will be given for the examination of Glucina, Yttria, and Oxide of Cerium.

§ 10. *GLUCINA*— $\text{Gl}^2 \text{O}^3$ —*Presence in the Mineral Kingdom.*

Glucina is not frequently found in nature ; it occurs only in silicious combinations,—it is found in Gadolinite, Leucophane, Phenakite,—which are the following :—

a. Silicate of Alumina and Glucina, as the *Emerald*, the *Beryl*—*Euclase*.

b. Basic Silicate of Alumina, with Alumina-Glucina ; as *Cymophane* and *Chrysoberyl* ; and

c. The Bisilicates of Glucina and Sesquioxide of Iron, with Sesquisilicate of Protoxides, and Oxy-sulphide of Manganese ; as *Helvine* [$3 (\text{Mn S}, \text{Mn O}) + 3 \text{Mn O}, 2 \text{Si O}^2 + \text{Gl}^2 \text{O}^3, 2 \text{Si O}^2 + \text{Fe}^2 \text{O}^3, 2 \text{Si O}^2$], from Schwarzenberg, in Saxony.

Examination for Glucina.

These glucinous minerals are so constituted, that the presence of glucina cannot be ascertained with certainty by their behavior to vitreous fluxes, because both alumina and silica, and the metallic oxides present in them, completely hinder the proper reaction of glucina, which, even when free from them, is not very striking. The metallic oxides, such as oxide of iron, oxide of

chromium, protoxide of manganese, and oxide of tin,—which last forms a constituent of the emerald, found in granular fragments at Broddbo and Finbo,—are easily detected, the first two by the colors which they produce with borax and microcosmic salt, the third by soda on platinum foil, and the last by reduction with soda.

The presence of glucina in metals is best ascertained in the moist way, as, for example, by the following method :—

Part of the mineral, very finely powdered, must be fused on charcoal, with a mixture of one and a half parts of soda and one of borax, in the reducing flame ; pulverized, dissolved in hydrochloric acid, evaporated to dryness ; the dry mass redissolved in water, the solution separated from the insoluble silica by filtration, and heated with a few drops of nitric acid, in order to convert the protoxide of iron into the sesquioxide, if it should happen to contain any. Ammonia is then to be added to the solution, until everything is precipitated. The precipitate is to be well washed, and heated with a solution of potassa. If it consists of alumina, glucina, sesquioxide of iron, oxide of chromium, and protoxide of manganese, the two earths will be dissolved out, while the metallic oxides remain undissolved and may be easily recognised, after being filtered, before the Blowpipe. The alkaline solution of alumina and glucina must be decomposed, either by boiling it moderately with chloride of ammonium, or by hydrochloric acid and ammonia, as has been already mentioned in speaking of the examination of silicates for alumina ; the precipitated earths are filtered from the liquid, and dissolved in a little hydrochloric acid and carbonate of ammonia, until a voluminous precipitate, which consists of alumina and glucina, is produced. As glucina is soluble in carbonate of ammonia, an excess of this reagent should be added, by which the precipitate diminishes, and the glucina redissolves. The mass should then be filtered, and the filtrate boiled ; during the ebullition the ammonia is expelled, and the glucina falls down in the state of a carbonate, which, after being separated by filtration, can be tested before the Blowpipe. It should show the reaction given in Table I. The alumina can also be tested before the Blowpipe with a solution of cobalt.

The presence of sulphur is detected in Helvine, as well as in the solution, in hydrochloric acid, of the mineral melted with soda and borax, by its evolving sulphide of hydrogen, which is readily distinguished by its smell, as also by a special examination for sulphur.

§ 11. YTTRIA—Y O—*Presence in the Mineral Kingdom.*

This earth occurs but seldom, though it is found in many different states of combination, as for example :—

a. As Diphosphate of Yttria, in Yttria-Phosphate or Phosphate of Yttria $[3 \text{ Y O}, \text{P O}_4]$;

b. As a combination of Fluoride of Calcium, with Fluoride of Yttrium, and Fluoride of Cerium, in variable proportions, in *Ytthro-Cerite* $[\text{Ca Fl} + \text{Y Fl} + \text{Ce Fl}]$;

c. As Titanate of Yttria, with Titanate of Zirconia, the Protoxides of Cerium, Manganese, and Iron, Lime, and other bases, in *Polymignite*, from Fredrikswärn ;

d. As Tribasic Tantalate of Yttria and Lime, with a perceptible quantity of Tribasic Tungstate of the Protoxide of Iron, in the *Black Ytthro-Tantalite*, from Finbo, Ytterby, and Kararfvet ; as Tribasic Columbate of Yttria and Lime, mixed with a little Tungstate of the Protoxide of Iron, in the *Dark Ytthro-Tantalite* from Ytterby ; and as Tribasic Columbate of Yttria and Oxide of Uranium, in *Yellow Ytthro-Tantalite*, from Ytterby and Finbo, $[3 \text{ Y O} \{ \frac{\text{T}_a \text{O}_3}{\text{U}_2 \text{O}_5} \}]$;

e. In Silicious combinations :—

¹ As Tribasic Silicate of Yttria, with Sexbasic Silicate of Cerium and Protoxide of Iron, in *Gadolinite*, from Ytterby, Finbo, and Broddbo, $[\frac{6 \text{ Fe O}, \text{Si O}_2}{6 \text{ Ce O}, \text{Si O}_2} + \frac{2 (3 \text{ Y O}, \text{Si O}_2)}{2 (3 \text{ Y O}, \text{Si O}_2)}]$;

² In a Silicious combination of Protoxide of Cerium, Alumina, Lime, Yttria, Protoxides of Iron, and Manganese, with chemically combined water, in *Orthite* ; and,

³ In a Silicious combination of Protoxide of Cerium, Yttria, Protoxide of Manganese, Alumina, and Lime, which at the same time contains carbon and water, in the *Pyrrorthite*. It is also found in Fergusonite, Euxenite, Aeschynite, Pyrochlore, Polykras,

Yttrotitanite. MOSANDER found Terbia and Erbia in various Yttrious minerals.

Examination for Yttria.

a. The phosphate of yttria can be easily recognised by its behavior before the Blowpipe, from its giving a regulus of phosphide of iron with boracic acid and iron, as will be subsequently mentioned under the head Phosphoric Acid, and by its being with difficulty dissolved by microcosmic salt.

But if it is required to detect with accuracy the presence of yttria itself, the assistance of the moist way must be had recourse to, and the following method pursued.

The mineral, in the state of a very fine powder, is first to be mixed with four times its weight of carbonate of soda, and the mixture fused in a platinum spoon, in the flame of the spirit lamp, until it ceases to effervesce. The fused mass, together with the spoon, should then be placed in some water, in a small porcelain vessel, and heated to the boiling point over the flame of the spirit lamp. The phosphate of soda formed, and the excess of carbonate of soda employed, are dissolved by the water, while the yttria, with some oxide of iron, which was contained in the mineral as basic phosphate of iron, remains behind undissolved. The residue separated from the fluid by filtration, is then edulcorated. Phosphoric acid may be detected in a small portion of the fluid, by the method which will be given under this acid.

The edulcorated residue of yttria and sesquioxide of iron must be dissolved while still moist, in a little hydrochloric acid, the solution diluted with water, one or two small spoonfuls of pulverized tartaric acid added, and the vessel allowed to stand in the heat until the acid is completely dissolved. According to ROSE, tartaric acid possesses the property of preventing the precipitation by alkalis, in any solutions of metallic oxides and earths; and to these yttria and oxide of iron belong. The solution is then saturated with ammonia, and sulphide of ammonium added till it ceases to throw down a black precipitate. The iron is thus separated as a sulphide, while the yttria remains unchanged in

the solution. If the supernatant liquid appears only yellow, when the precipitate has sunk completely to the bottom, it should be filtered; but if green, the whole should be first allowed to digest in a very gentle heat until it becomes yellow. The sulphide of iron, after its separation by filtration, may be roasted on charcoal, and the resulting black or magnetic oxide, treated with borax. The filtered solution should be evaporated to dryness, and the dry mass ignited in a platinum spoon over the spirit lamp, until all the charcoal of the tartaric acid is consumed, and the yttria appears on cooling perfectly white. The dried mass should be placed in the spoon by degrees, as, during the decomposition of the acid, a portion of the yttria may be lost, by being projected out of the recipient.

The yttria obtained in this operation should behave itself before the Blowpipe in the manner described in Table I.

Sesquioxide of iron may be separated in another way from yttria, by employing the subsequent method. Rather more than the necessary quantity of carbonate of baryta should be gradually added to a cold solution of yttria and sesquioxide of iron, in hydrochloric acid, and the fluid stirred with a glass rod, so long as carbonic acid gas is evolved; the sesquioxide of iron is precipitated, while the yttria remains in the solution. The supernatant liquid is then separated from the precipitate by filtration, the baryta thrown down by dilute sulphuric acid, and the yttria by potassa or ammonia.

b. Ytthro-cerite is readily recognized before the Blowpipe, but its yttria cannot be detected by this means alone.

The Ytthro-cerite from Finbo, when heated in a flask, yields some water, which has an empyreumatic color. The dark colored variety loses its color by this treatment, and becomes white.

It is infusible when heated on charcoal *per se*; with gypsum it fuses to a bead, which does not become clear, however, at any temperature.

It readily fuses with borax and microcosmic salt to a transparent yellow glass, which is colorless when cold, but at a certain point of saturation translucent.

With soda, unless too much be used, it melts on charcoal to a

clear glass, which upon cooling, and also when the blast is directed on it for a long time, becomes turbid ; a larger addition of soda converts it into a difficultly fusible enamel, which remains on the charcoal while the excess of soda is absorbed.

The Ytthro-cerite from Broddbo decrepitates but slightly when heated in a flask. It is infusible on charcoal, but on heating it becomes first milk-white, then brick-red, but, however, always unequally colored. It does not fuse with gypsum.

It behaves to borax and microcosmic salt in a nearly similar manner to oxide of cerium, as it contains a large quantity of fluoride of cerium mixed with it.

It is acted upon by soda, tumefies a little, but is not dissolved. The greater part of the soda is absorbed by the charcoal, leaving a grey scoria behind.

If it be required to separate the yttria from Ytthro-cerite, it must be done in the moist way, according to the following method ; the finely powdered mineral is to be dissolved in hydrochloric acid, heated to ebullition, the solution evaporated nearly to dryness, diluted with water, and filtered in case any undissolved particles are observed. From this diluted solution, alumina, yttria, and protoxide of cerium may be precipitated by an excess of ammonia ; the whole should then be warmed, and the precipitate separated by filtration. Lime is precipitated from the filtrate by oxalate of ammonia. The precipitate obtained by ammonia should be edulcorated with hot water, until the water which percolates through the filter is no longer rendered turbid by oxalate of ammonia, and digested at a moderate heat while still moist with caustic potassa, in order to separate the alumina from the yttria and protoxide of cerium. When the digestion is complete, the alkaline solution is to be diluted with water, and filtered. The alumina may be separated from the filtered solution slightly heated, by the necessary quantity of chloride of ammonium, or by ammonia from the solution, rendered acid by hydrochloric acid. In both cases, the precipitate after filtration must be well edulcorated, and then tested with a cobalt solution. The undissolved yttria and protoxide of cerium is to be well washed with hot water, dissolved in hydrochloric acid, and the

solution diluted with water. A crust of crystallized sulphate of potassa must then be placed on the surface of the liquid, and the whole allowed to stand about twenty-four hours. During this time the solution will become saturated with sulphate of potassa, with which the yttria and protoxide of cerium will combine, forming double salts ; of which the one formed with yttria is soluble in a saturated solution of sulphate of potassa, while that formed with protoxide of cerium, on the other hand, is insoluble, and falls to the bottom as a white amorphous powder. After the lapse of the above mentioned time, the remaining crust is removed, the precipitate separated by filtration, and edulcorated with a saturated solution of sulphate of potassa. After the edulcoration, it is to be dissolved in boiling water, the protoxide of cerium precipitated from the still warm solution by potassa, filtered, the precipitate edulcorated, and ignited in the platinum spoon. During the ignition it is converted into sesquioxide, and becomes of a cinnamon brown color. The yttria which remains in solution is also to be thrown down from the warm solution by potassa, filtered, and ignited in the platinum spoon. It must possess a white color after the ignition ; if it becomes brown, it contains sesquioxide of iron, which can be separated after the earth has been dissolved in hydrochloric acid, by the method already given for phosphate of yttria. A separate examination is required for the detection of hydrofluoric acid, which will be subsequently given.

c. Polymignite,—the name of which is derived from *πολυς* much, and *μυρον*, I mix,—according to BERZELIUS, behaves in the following manner.

Heated in a bulb tube, it yields no water ; it is infusible on charcoal, and does not change its color or brilliancy. Borax readily dissolves it to an iron colored glass, which, by a larger addition, acquires the property of becoming streaked with turbid lines, by which it generally appears of a brownish-yellow color. From a larger addition, it becomes turbid *per se*, on cooling ; with tin the bead becomes of a red color, passing into yellow.

It is difficultly dissolved by microcosmic salt, the glass assuming a reddish color in the oxidating flame, which is not altered

on the addition of tin. It is decomposed by soda, becoming greyish-red, but does not fuse ; it gives the same reaction as manganese on platinum foil.

In order to separate the yttria, the moist way must be had recourse to, as in the case of the first two minerals. The method of procedure is as follows :—The finely powdered mineral is to be fused with from six to eight times its weight of bisulphate of potassa, in a platinum spoon, and the fused mass digested with a sufficient quantity of water, until the whole is dissolved out of the spoon ; the sulphates of the protoxides of iron, cerium, and manganese, of yttria, lime, and magnesia, are dissolved, while the titanio acid, in combination with zirconia, and a part of the protoxide of iron, remains behind ; the whole must then be kept for some time at a moderate ebullition, and filtered. The protoxide of cerium may be separated from the solution by a piece of sulphate of potassa, in the manner already described under Yttrocercite. To the solution, from which the double salt has been separated by filtration, a few drops of nitric acid are to be added, in order to convert the protoxide of iron into the sesquioxide ; a little chloride of ammonium should also be added, to prevent the precipitation of protoxide of manganese and magnesia, if it should contain them, and the yttria and sesquioxide of iron thrown down with ammonia. Lime is separated from the filtered solution, by oxalate of ammonia ; and magnesia and protoxide of manganese, by microcosmic salt. The precipitate obtained by ammonia must be dissolved in hydrochloric acid, and the sesquioxide of iron separated from the yttria, by the method already given for the decomposition of phosphate of yttria.

d. The following has been found to be the behavior of Yttrotantalite before the Blowpipe :—Heated *per se*, in a bulb tube, it gives off water, and, if previously black, becomes yellow. Some specimens become mottled with dark spots, which remain unchanged in the heat ; they become white on ignition, while the upper part of the glass is attacked. The water eliminated first colors brazil-wood paper yellow, and then bleaches it.

Borax dissolves it to an almost colorless glass, which, by a larger addition, becomes turbid on cooling.

It is at first decomposed by microcosmic salt, the tantalic acid remaining undissolved as a white skeleton, which fuses, however, in a strong blast. The black variety of Yttro-tantalite, from Ytterby, produces a glass which becomes of a light rose color by a strong reducing flame, on cooling, derived from the presence of tungsten. The dark and yellow varieties from Ytterby, become, on cooling, of a slight but beautiful green color, from the presence of uranium. The Yttro-tantalite, from Finbo and Kararfvet, gives a strong ferruginous tint, which becomes darker when the test for oxide of uranium is employed.

It is decomposed without solution by soda. It exhibits on platinum foil the presence of manganese. Traces of tin are obtained by reducing with soda and borax; the Finbo variety, however, contains so much iron that the tin cannot be recognized.

The preparation of yttria from Yttro-tantalite is attended with much difficulty. The separation can only be performed in the moist way, and safest when more than 100 milligrammes of the mineral are employed, for which larger vessels are required than the Blowpipe operator can conveniently carry about with him.

e. Silicious combinations :—

¹ Gadolinite, from Ytterby, Finbo, and Broddbo ;—

BERZELIUS gives the following account of their behavior before the Blowpipe. These Gadolinites are of two varieties, one of which, α , is of such a vitreous nature that it appears like black glass; the other, β , on the contrary, has a splintery fracture, and not so conchoidal; the former contains no glucina, but it is very probable that the latter has some per cents.

Variety α , *per se*, in a bulb tube, undergoes no change, and gives off no water; if it be heated nearly to the point of fusion, it appears to glow for an instant, as if it had caught fire; it tumefies a little, and if the fragment be large, fissures appear here and there in it, and the color assumes a light greyish-green; no volatile matter is, however, given off. The same phenomena take place on charcoal; it does not fuse, but in a strong heat it becomes black on the edges.

Variety β , *per se*, swells out into dendritic ramifications, and

becomes white, at the same time giving off moisture. The above mentioned luminous appearance is very rarely observed. In all other points both behave alike to fluxes.

It is readily dissolved by borax, forming an intense ferruginous-colored glass, which becomes of a deep bottle-green in the reducing flame.

It is difficultly dissolved by microcosmic salt. The glass assumes a ferruginous color, and the fragment employed becomes rounded on the edges, but it remains white and opaque, so that the silicic acid cannot be separated from the phosphoric acid; by this it is principally distinguished from the Kararfvet variety. Soda dissolves it to a reddish-brown semifused scoria. The variety β melts into a bead with soda, when too large a quantity of the flux is not employed. None of them exhibit the slightest trace of manganese on platinum foil.

The moist way must be had recourse to, in order to separate yttria from Gadolinite. The method of procedure is as follows:—

The finely powdered mineral is to be fused to a bead, with an equal bulk of carbonate of soda and borax, on charcoal, in the oxidating flame; the bead pulverized, the powder dissolved in hydrochloric acid, the solution evaporated to dryness, the dry mass dissolved and filtered from the insoluble silicic acid. A few drops of nitric acid are then to be added to the solution, and the whole heated, in order to convert the protoxide of iron into the sesquioxide, and the yttria, protoxide of cerium, sesquioxide of iron, and glucina, when present, thrown down by ammonia. Lime may be precipitated from the supernatant liquid, when present, by oxalate of ammonia. The precipitate by ammonia, after edulcoration with hot water, must be treated in the heat while still moist, with a solution of caustic potassa, in order to separate any glucina present; the alkaline solution should then be diluted with water, and the glucina separated from it by chloride of ammonium, in the way already given under the head Glucina. The residue, after edulcoration, is then to be dissolved while still moist, in a small quantity of hydrochloric acid, and the protoxide of cerium separated by sulphate of potassa, by the method given

for the decomposition of Yttrio-cerite. The sesquioxide of iron and the yttria are precipitated by a solution of potassa ; the precipitate, afteredulcoration, dissolved in hydrochloric acid, and both constituents separated by the method given for the analysis of Phosphate of Yttria.

² Orthite, and Pyrorthite, contain only from three to four per cent. of yttria ; it cannot be separated in a pure state from these minerals in the dry way, and only by the moist way when a large quantity—as 100 milligrammes—is employed.

The behavior of both these minerals before the Blowpipe is as follows :—

Orthite, from Finbo and Gottliebsgang, as well as that from Granit, near Stockholm, and Söderköping :—

Per se, in a bulb tube, it yields water, and at a high temperature assumes a light color. It tumefies and becomes yellowish-brown on charcoal, and at length fuses, under ebullition, to a black blebby glass.

It is easily dissolved by borax ; the glass becomes blood-red in the reducing flame when hot, and yellow on cooling. In the reducing flame it becomes of a ferruginous green.

It is decomposed, with the usual appearances, by microcosmic salt.

With soda it tumefies ; with a very small quantity it fuses, but with a large quantity it intumesces to a greyish-yellow recement. On platinum foil it shows the presence of manganese.

Pyrorthite from Kararfvet :—

Per se, in a bulb tube, it at first gives off a large quantity of water, which towards the end becomes yellow, and has an empyreumatic odor, the mineral becoming coal black. Moderately heated on charcoal, and then ignited at a single point, it begins to become luminous, and afterwards, of itself, continues to glow without flame or smoke. If a number of small fragments be laid together, or a small heap of coarse powder be taken, the combustion proceeds better ; by a moderate heat it is also increased. When the combustion is ended, the assay is white, or greyish-white ; with different pieces this is not always the same, and

sometimes it even passes into red. The fragments are now so light, that they cannot be kept on the charcoal in the blast. Held in the forceps, they fuse with difficulty on the surface to a black dull bead.

With borax it fuses readily to a glass, which behaves similarly to the borax glass of Orthite.

It is with difficulty dissolved by microcosmic salt. The porous fragment remains on the surface of the bead, so long as the mass is molten, but on cooling it is absorbed; but if the bead be again heated, it comes to the surface as before.

Its comportment with soda is similar to that of Orthite.

§ 12. ZIRCONIA— $Zr^2 O^3$ —*Presence in the Mineral Kingdom.*

This earth is found in combination with Silicic and Titanic Acids, namely:—

a. As Silicate of Zirconia, in the *Zircon* and *Hyacinth* [$Zr^2 O^3$, $Si O^2$];

b. In a combination of Sesquibasic Silicates of Soda and Lime, with Silicates of the Sesquioxide of Iron, Manganese, and Zirconia, also Chloride of Sodium, in *Eudyalite* [$Na Cl + \{ \frac{2}{3} Ca O, \frac{2}{3} Si O^2 \} + \{ Zr^2 O^3, \frac{2}{3} Si O^2 \}$];

c. In a combination of the Titanates of Zirconia, Yttria, Protoxides of Cerium, Manganese, and Iron, Lime, and other bases, in *Polymignite*, also in *Oerstedtite*, *Polykras*, *et cetera*.

Zirconia occurs also with Tantallic Acid in *Wöhlerite*, *Pyrochlore*, *Fergusonite*.

Examination for Zirconia.

Zirconia cannot be detected by the aid of the Blowpipe alone, either in those minerals where it forms only a secondary constituent, or even in those where it forms the primary ingredient; but the minerals themselves may be immediately recognized by their behavior before it.

a. The Zircon and Hyacinth from Ceylon, Finbo, Fredrikswärn, and Expailly, behave themselves, as under :—

Per se, the colorless transparent varieties remain unchanged. The clear red Hyacinth loses its color, and becomes either perfectly limpid, or very slightly yellow. The opalescent brown from Fredrikswärn loses its color, and becomes white, similar to fractured glass. The dark variety from Finbo gives off a little moisture, becomes milk white, and appears scopiform. None of them can be fused, either in the state of powder or in splinters.

Zircon is fused with difficulty to a clear glass by borax, which, at a certain saturation, becomes streaked with turbid lines, and from a still greater quantity, becomes translucent on cooling.

Zircon remains intact with microcosmic salt ; a fragment subjected to its action retains the sharpness of its edges, and when Zircon powder is employed, it is so little changed that it cannot be determined whether it was attacked or not.

The glass remains perfectly colorless or milk white when the powder is added to it, as well in the oxidating as in the reducing flame.

It is not fused with carbonate of soda ; the soda, however, attacks it slightly on the edges, but it afterwards sinks into the charcoal. Most specimens of Zircon show the presence of manganese, when treated on platinum foil.

b. Eudyalite exhibits the following behavior before the Blow-pipe :—

In a bulb tube it gives off moisture ; it readily fuses on charcoal to a greyish glass, passing into green.

It is readily dissolved by borax, to a glass slightly colored by iron, which does not become turbidly streaked.

It is easily decomposed by microcosmic salt ; the silicious skeleton swells up so much, that the bead generally loses its form. By this peculiarity it distinguishes itself from the garnets, to which it is otherwise similar.

Soda dissolves it to a glass, which is not very fluid ; with more soda, it sinks into the charcoal. It shows the presence of manganese on platinum foil.

The comportment of Polymignite before the Blowpipe has been already given under Yttria—see page 144. The assistance of the moist way must be had recourse to, if it be desired to separate zirconia from the Zircon, Hyacinth, or Eudyalite, with the Blowpipe, the method of procedure being as follows :—The mineral, in the state of a very fine powder, is first fused to a glass, for the Zircon, with a mixture of one and a half parts of soda, and three parts of borax, to one of the mineral ; and for Eudyalite, two parts of soda, and one and a half parts of borax, on charcoal, in the oxidating flame ; the glass pulverized, the powder treated with hydrochloric acid, and the solution evaporated to dryness, in order to be able to perfectly separate the silica. The evaporation should not be effected too rapidly, nor at too strong a heat, as in that case a portion of the zirconia would remain undissolved with the silica. The dry mass thus obtained is then to be treated with a sufficient quantity of water, and the silicic acid separated by filtration. If the mineral should happen to contain iron, as is the case with Eudyalite, the greater part of it will be found in the solution as protoxide, which should be converted into sesquioxide, by the addition of a few drops of nitric acid. When manganese is present, it will also be in the solution as protoxide, and should be rendered incapable of precipitation by ammonia, by the addition of a little chloride of ammonium.

Zirconia and sesquioxide of iron should then be precipitated by ammonia, the whole heated to ebullition, in order that the precipitate, which is very voluminous, may collect, and thus be more easily filtered. If the mineral contains lime, it remains behind in the solution, and may be precipitated by oxalate of ammonia.

Nothing now remains to be done, but to separate the sesquioxide of iron from the zirconia, which can be performed in two ways :—

1. The precipitate is welledulcorated with boiling water, dried, ignited in a platinum spoon, and treated with hydrochloric acid. The sesquioxide is by this means almost perfectly dissolved, while the zirconia, which after ignition is insoluble in this acid, remains nearly pure intact.

2. The precipitate, while still moist, is dissolved in hydrochloric

acid, and the oxide of iron precipitated by sulphide of ammonium, as in the method given at page 141, for the separation of oxide of iron and yttria. The liquid filtered from the sulphide of iron is to be evaporated to dryness, and the dry mass ignited in a platinum spoon, over the spirit-lamp, until the whole of the charcoal of the tartaric acid is burned off, and the zirconia appears perfectly white.

A much more simple method may be employed for the detection of zirconia, in the Zircon or Hyacinth, as they contain no lime. The solution is first neutralized in a test-glass with potassa, and a crystal of sulphate of potassa laid in it, of such a size that it is sufficient to perfectly saturate the solution, and the whole warmed. As soon as a portion of the crystal has dissolved, a double compound of sulphate of zirconia and potassa forms, which falls to the bottom of the glass as a flocculent basic salt, and when a sufficient quantity of the crystal has dissolved, the whole of the zirconia is precipitated in this state, and can be obtained by filtration; it should then be welledulcorated, and boiled with caustic potassa, by which it is decomposed, leaving pure hydrate of zirconia behind.

Zirconia cannot be obtained, with successful results, from even a quantity of 100 milligrammes of Polymignite, owing to the presence of titanitic acid, to which zirconia is in many respects similar.

§ 13. *THORINA*—Th O—*Present in the Mineral Kingdom.*

The density of this earth, according to GRAHAM, is 9.402, and it is therefore superior to all others. It is found in combination with silicic acid, in a rare mineral, *Thorite* [$3 \text{ Th O}, \text{Si O}_2 + 6 \text{ aq}$]; which has been analyzed by BERZELIUS only. It contains 57.91 per cent. of Thorina, besides Lime, Magnesia, Oxides of Iron, Manganese, Uranium, Tin, Silica, Alumina, Potassa, Soda, and Water.

Professor WÖHLER has since detected this earth in small quantity—3 per cent.—in *Pyrochlore*, from the Ural mountains.

Examination for Thorina—or Thoria.

Owing to the scarcity of Thorite, an opportunity has not been afforded of examining a specimen for Thorina before the Blowpipe. The following is the humid method given by BERZELIUS :—

The finely powdered mineral is digested with hydrochloric acid ; when the whole mass, with disengagement of chlorine, becomes yellow and gelatinous, and must be evaporated to dryness in a water bath. The dried mass is then to be treated with water, filtered, and sulphide of hydrogen passed through the liquid, by which some sulphides of lead and tin are thrown down. It is again filtered, the thorina precipitated with caustic ammonia, the precipitate filtered and edulcorated. It is now rendered impure only by the oxides of iron, manganese, and uranium. To free it from these, the precipitate is to be dissolved, while still moist, in dilute sulphuric acid, and the solution evaporated until very little of it remains. During the evaporation, a white slightly cohering soft mass is deposited in abundance ; this is neutral sulphate of thorina. It has the curious property of being very slightly soluble in hot, particularly so in boiling water. The acid is then decanted off, the white salt thrown on a filter and edulcorated from the adhering mother liquor, dried, and strongly ignited, by which the earth is obtained pure and white. The decanted acid liquor, and the water of edulcoration, as they contain more thorina, are then to be evaporated to a small bulk, saturated with carbonate of potassa, and mixed with a boiling saturated solution of sulphate of potassa. On cooling, the excess of sulphate of potassa, with a double salt of sulphate of thorina and potassa, which is perfectly insoluble in a saturated solution of the former salt, crystallize out of the liquid. It is then washed with a solution of sulphate of potassa, dissolved in warm water, and precipitated with ammonia. The precipitate yields, on ignition, an earth slightly tinged yellow by a trace of manganese, which may be purified by treating it as in the first method. It is obtained perfectly isolated only after ignition.

β. EXAMINATION OF METALS AND THEIR OXIDES.

§ 1. CERIUM—Ce—LANTHANUM—La—and DIDYMIUM—D —Presence in the Mineral Kingdom.

Cerium belongs to the rare metals, and is found in the following minerals, nearly always in company with more or less Lanthanium and Didymium :—

a. With Fluorine, in neutral *Fluoride of Cerium* from Broddbo, Ce Fl mixed with $\text{Ce}^2 \text{Fl}^3$, besides Yttria and Water ; in basic *Fluoride of Cerium* from Finbo [$\text{Ce}^2 \text{Fl}^3 + 3 \text{Ce}^2 \text{O}^2, \text{H O}$] ; in *Fluoride of Cerium* from Rytterhyttan [$(\text{Ce}^2 \text{Fl}^3 + 3 \text{H O}) + \text{Ce}^2 \text{O}^2, \text{H O}$] ; in *Fluoride of Cerium* with *Fluoride of Yttrium* from Finbo, Ce Fl, Y Fl, mixed with Silicic Acid ; in *Ytthro-Cerite* with Yttria and Lime ;

b. In combination with Phosphoric Acid, in *Cryptolite* $3 \text{Ce O}, \text{P O}^5$ together with a little oxide of Iron ; in *Monazitoid* with Lanthanium, Lime, and Water ; in *Monazite* with a mixture of Lime, Magnesia, and Binoxide of Tin ;

c. As Protoxide with Carbonic Acid, in *Carbonate of Cerium* [$\text{Ce O}, \text{C O}^2$] ; in *Parisite* with Lanthanium, Didymium, Lime, *et cetera* ;

d. As Protoxide in Titanic Acid combinations, namely, with Lime, Thorina, Protoxides of Uranium, Iron, and Manganese, in *Pyrochlore* ; and with Zirconia, Yttria, Lime, Protoxides of Manganese and Iron, and other bases, in *Polymignite* ;

e. As Protoxide, in Silicious combinations, namely :—Singly, in *Cerite* [$3 \text{Ce O}, \text{Si O}^2 + 3 \text{aq}$] ; with Protoxide of Iron, Lime, and Alumina, in *Cerine* and *Allanite* ; with Yttria and Protoxide of Iron, in *Gadolinite* ; with Protoxide of Iron, Yttria, Lime, Protoxide of Manganese, and Water, in *Orthite* ; and with Yttria, Protoxides of Iron and Manganese, Alumina, Lime, together with a little Carbon and Water, in *Pyrrorthite*. It is also found in Aeschynite, Fergusonite, Euxenite, Polykras, Bodenite, Tschewkinite, Mosandrite, and Tritomite.

Examination for Cerium, Lanthanum, and Didymium.

In minerals, in which no other coloring matter, or at most only traces of such exist, besides protoxide of cerium, as in Fluoride of Cerium, Yttrio-Cerite, Carbonate of Cerium, and Cerite, the cerium is readily detected with fluxes. Red or dark yellow beads are obtained with borax and microcosmic salt in the oxidating flame, according as a large or small quantity has been dissolved, and the mineral contains a large or a small proportion of protoxide of cerium ; these beads, on cooling, and also when subjected to the reducing flame, lose their color to such a degree, that the bead of microcosmic salt becomes perfectly colorless. If the mineral contains a slight trace of iron, the borax bead becomes greenish in the reducing flame.

Protoxide of cerium cannot be recognized in this way, in such minerals as contain at the same time protoxide of iron, uranium, or titanous acid, as, for instance, Pyrochlore, Polymignite, Cerine, Allanite, Gadolinite, Orthite, and Pyrorthite. The operator succeeds better with such minerals, by melting them in a finely powdered state, with from six to eight times their bulk of bisulphate of potassa, dissolving the fused mass in a large quantity of water, and boiling the solution for some time, in order to precipitate titanous acid, if the mineral contains any. If the substance contains a large quantity of titanous acid, and at the same time zirconia, and protoxide of iron, the titanous acid, with the zirconia, and a part of the protoxide of iron, remain behind. If the precipitation of the titanous acid takes place, it is to be separated by filtration, and the protoxide of cerium thrown down from the filtered solution, in the manner described at page 144 ; or, in a far shorter way, as follows :—

First, the fluid is boiled for a short time in a test tube ; if it remains clear, the whole of the titanous acid has been separated ; when this is the case, a crystal of sulphate of potassa, rather more than sufficient to saturate the liquid, must be suspended in it, and the whole heated anew. If the solution contains protoxide of cerium, it falls of a white color, as a double salt of sul-

phate of potassa and cerium, as soon as the solution becomes saturated with the sulphate of potassa. This occurs even when the quantity of cerium present is exceedingly small, the solution at first being only slightly turbid, but subsequently, on allowing the vessel to stand undisturbed for some time, the undissolved portion of the sulphate of potassa becomes covered with a white light friable powder. If the mineral contains zirconia, without titanitic acid, it will be thrown down along with the cerium double salt, as basic sulphate of zirconia and potassa, unless, in the commencement, care be taken to remove it from the aqueous solution of the fused mass. Thorina, in case it is present, will also be thrown down with the cerium. When lime is present in the mineral, a portion of it is dissolved in the treatment of the fused mass with water, as sulphate of lime, and is also separated by the sulphate of potassa. The resulting precipitate should therefore, after separation by filtration, be well edulcorated, and tested for cerium with borax; the double salt is by this means decomposed, the sulphuric acid and potassa separate as sulphate of potassa, and flow down the platinum wire, if the end with the loop be held higher than the other. The borax bead remains clear, provided it is not saturated, and by it a very small trace of oxide of cerium may be recognized, by the color given at page 90, Table II.

Before the Blowpipe the oxide of lanthanum behaves in the following manner:—It does not fuse with soda, which is absorbed by the charcoal, leaving the oxide behind as a dirty white mass. Borax dissolves it in considerable quantity. The bead is perfectly colorless, both while hot and after cooling, and it is only when it has been saturated to excess with oxide of lanthanum that the bead appears on cooling of a light rose color, which is rendered perceptible by holding it against a white ground; flaming will not cause its opacity.

When the bead appears, while hot, of a yellow color, it is a sign that the oxide of lanthanum still contains oxide of cerium, in which case it may also be rendered opaque by flaming. Its behavior with microcosmic salt is as with borax.

In the year 1839, MOSANDER found that the oxide of cerium, which up to that time was supposed to contain only one metal, was

mixed with another, which he called Lanthanium, a name derived from the Greek word synonymous with *lying concealed*. In another research upon the oxide of cerium in 1842, he found that there was a third oxide in the compound, and he called the metal of this oxide Didymium, from the Greek meaning *twins*.

For separating the oxide of cerium from the oxide of lanthanum, MOSANDER has given two methods. According to one, the heated oxides are treated with dilute nitric acid, containing one part of acid to fifty parts of water, by which the oxide of lanthanum is dissolved, leaving the oxide of cerium nearly pure. By the other method, the mixed oxides are to be dissolved in hydrochloric acid, and potassa in excess added to the solution, which throws down the hydrated oxides; this precipitate is filtered off, suspended in caustic potassa in an appropriate flask or beaker, and chlorine gas transmitted through the menstruum, by which means the oxide of lanthanum is dissolved, while the oxide of cerium remains in the form of a heavy yellow or yellowish-red powder. If the whole of the oxide of cerium be not separated by this treatment, the operation of adding hydrochloric acid, followed by precipitation with caustic potassa, and the transmission of chlorine through the liquor holding the precipitate in suspension, must be repeated as above till the whole is removed.

Impure oxide of cerium is dissolved by sulphuric acid diluted with its weight of water; the solution is then mixed with fifty parts of pure water, and the liquid made to boil. During ebullition the solution becomes opaque, and deposits a sulphur-yellow precipitate which is perfectly pure basic sulphate of cerium. All the oxide of lanthanum which was mixed with the oxide of cerium, remains in the solution, which also contains a large quantity of the latter element. To obtain the last portions of the oxide of cerium, caustic soda is added to the acid liquor, and the precipitate which it occasions filtered off, washed, and dissolved in nitric acid, the solution evaporated, and the residuary salt ignited, after which the oxide of lanthanum is dissolved out by nitric acid. The residual oxide of cerium is then treated with sulphuric acid, and the solution diluted and boiled as above, when a precipitate of basic sulphate of cerium falls.

According to SCHREER, the oxide of cerium may be entirely freed from the last traces of the oxides of lanthanum and didymium, by dissolving in hydrochloric acid, neutralizing very cautiously with ammonia, adding acetate of ammonia, and then oxalate of the same alkali ; the resulting oxalate of the oxide of cerium collected upon a double filter, washed, dried, ignited, and again treated with very weak nitric acid, yields pure oxide of cerium.

The oxide of didymium is the cause of the brown color of the oxide of cerium, and the reddish appearance is due to the oxide of lanthanum. Pure oxide of cerium is citron-yellow ; that of lanthanum, white ; and that of didymium, brownish-yellow.

MOSANDER found that the compounds of didymium were in many respects analogous to those of manganese : the oxide is brown, the sulphate of the oxide amethystine : its oxide gives a brown color to oxide of cerium, and a red tinge to the salts of yttria.

In many of the forementioned minerals in which, besides oxide of cerium there are oxides of lanthanum and didymium, and no other colored oxides to any appreciable extent, *exempli gratia*, in fluoride of cerium, in phosphatic and carbonated combinations, and in Cerite, the presence of the oxides under consideration, is easily detected. In the oxidating flame with borax or microcosmic salt these minerals afford a red or dark-yellow bead, according to the quantity of the assay which may be dissolved in the glass, but the beads with the latter reagent become colorless in the cold, or under the influence of the reducing flame, while the borax bead under the same circumstances becomes streaked or enamelled, according to the amount of silicic acid in the mineral.

HERMANN states that he could not detect the least trace of didymium in his researches on Cerite.

L. BONAPARTE had been occupied for some time in the chemical investigation of several metallic valerianates, and especially those of cerium, when he became aware of MOSANDER's discovery of didymium. He had succeeded in finding in a concentrated solution of valerianic acid a most excellent means of separating the binoxide of cerium in a pure state from the binoxide of didymium.

Valerianic acid possesses a singular and unexpected affinity for binoxide of cerium, and occasions an abundant precipitate in a neutral and concentrated solution of the mixed nitrates of the binoxides of didymium and cerium. The yellowish-white precipitate consists entirely of the valerianate of the binoxide of cerium, and to obtain the pure binoxide it only requires a thorough washing and calcination at a strong red heat in contact with the atmosphere. This oxide is of a very pale yellow color, like that described by MOSANDER, who, however, expressly states that he had hitherto not been able to find any absolute means of separation for the oxides of cerium, lanthanum, and didymium.

The oxide of didymium remains dissolved in the acid liquid, from which the valerianate of the binoxide of cerium has been precipitated. A portion of the cerium, however, remains mixed with the didymium, for the valerianates of these two metals are slightly soluble in water, and still more so in acid liquids, especially that of didymium, which is far more soluble in weak acids than that of cerium. It is possible, however, by means of valerianic acid, to obtain the oxide of didymium in a state of purity, although with much more difficulty than that of cerium.

BONAPARTE further states that, to obtain the pure valerianate of the binoxide of cerium from the mixed nitrates of the binoxides of cerium and didymium, it is necessary to throw down this salt by an aqueous and concentrated solution of valerianic acid; if a soluble valerianate were employed, the didymium, which is very slightly soluble in the state of valerianate in neutral solutions, would likewise be precipitated. The easy preparation of the binoxide of cerium in a pure state is owing, therefore, to the great solubility of valerianate of didymium in acid liquids, and to the less solubility of that of the binoxide of cerium under similar circumstances.

In minerals which contain oxides of uranium and iron, titanitic acid, *et cetera*, the presence of oxides of cerium, lanthanum, and didymium are not easily recognized by the Blowpipe. When the least doubt arises, the best way is to have recourse to the humid method, and to treat the oxides thus obtained with the special fluxes.

§ 2. *MANGANESE—Mn—Presence of this Metal in the Mineral Kingdom, and in the Scoria from Smelting Works.*

Manganese occurs in nature :—

- a. As a Sulphide, in *Mangan-Blende* [Mn S] ;
- b. As an Oxide, alone, as well as in combination with other Metallic Oxides ; *exempli gratia, per se* in *Grey Oxide of Manganese* [Mn O^2] ; as a Hydrate, in *Black Wad* [$\text{Ba O, Mn}^2 \text{O}^3, \text{H O}$] and [$\text{Mn}^2 \text{O}^3, \text{H O} + 2 (\text{Fe}^2 \text{O}^3, \text{H O})$] ,

A sample of the latter, analyzed by WACKENBODEB, gave,—

Sesquioxide of Manganese	32·73
Peroxide of Lead	12·33
Protoxide of Lead	8·0
Sesquioxide of Iron	9·33
Oxide of Copper	4·0
Oxide of Cerium	0·33
Silicic Acid	0·13
Quartz	2·60
Water	31·33
	<hr/>
	100·78

With Oxide of Cobalt and Water, in *Black Earthy Cobalt* [$\text{Co O, Mn}^2 \text{O}^3 + 3 \text{ aq}$] ;—according to DÖBEREINER's analysis, the formula is [$\text{Co}^2 \text{O}^3, \text{Mn}^2 \text{O}^3 + 3 \text{ aq}$]—and with Sesquioxide of Iron, Protoxide of Iron, and Oxide of Zinc, in *Franklinite—Dodecahedral Iron Ore*,— $\left[\begin{smallmatrix} \text{Mn} & \text{O} \\ \text{Fe} & \text{O} \\ \text{Zn} & \text{O} \end{smallmatrix} \right] \text{Mn}^2 \text{O}^3$; or, according to KOBELL, [$2 \text{ Zn O, 3 Mn O}^2 + 5 (\text{Fe O, Fe}^2 \text{O}^3)$] ;

c. As a Protoxide, in a Phosphoric Acid combination containing Protoxide of Iron and a small quantity of Lime ; *exempli gratia, Triplite—Phosphate of Manganese and Iron*— $[4 \text{ Mn O, P O}^5 + 4 \text{ Fe O, P O}^5]$;

d. As Protoxide, with Tungstic Acid and Tungstate of Iron, in *Wolfram* [$\text{Mn O, W O}^3 + 3 (\text{Fe O, W O}^3)$] ;

e. As Protoxide, with Tantallic Acid, Protoxide of Iron, Oxide of Tin, Tungstic Acid, and Lime, in various *Tantalites* ;

f. In an oxidized state, in Silicic Acid compounds ; *per se*, in *Red Siliciferous Oxide of Manganese* [3 Mn O , Si O^2] ; further, with Protoxide of Iron, Alumina, and Water, in *Umbur* ; with Protoxide of Iron, Protochloride of Iron and Water, *et cetera*, in *Pyrosmalite* [$\text{Fe}^2 \text{ Cl}^3 + \text{Fe}^2 \text{ O}^3$, 6 H O] or [$4 (3 \text{ Fe O}$, $2 \text{ Si O}^2 + 3 \text{ Mn O}$, $2 \text{ Si O}^2)$] ; with Alumina, Lime, and sometimes

Magnesia, in *Garnet* ;—general formula $\left[\begin{smallmatrix} 3 \text{ Mg O} \\ 3 \text{ Fe O} \\ 3 \text{ Ca O} \\ 3 \text{ Mn O} \end{smallmatrix} \right] \text{Si O}^2 + \text{Al}^3 \text{ O}^3$, Si O^2] ; with Protoxide of Iron, Alumina, and Water, in *Chloropal* ; and also as an extremely small ingredient of numerous other Silicates.

Manganese is very seldom met with in the metallic state, in the scorise or recrements from the smelting process. It is generally in combination with oxygen, sulphur, or other metals, or else is taken up by the silicates which mostly compose the slags. Manganese also occurs in Manganglance, Hausmanite, Braunitz. Manganite, Psilomelan, Varvicite, Polianite, Pyrolusite, Grovoville, Huraulite, Eisenapatite, Hetopozite, Triphyline, Tetraphyline, Manganocalcite, Tantalite—from Kimito, Tamela, Finbo, Broddbo, and Bodenmais—Tephroite, Fowlerite, Troostite, Knebelite, Helvine, Karpholite.

Examination for Manganese.

Substances containing no other metals save manganese, give colored glasses with borax and microcosmic salt in the oxidating flame, and can be very readily recognized, when dissolved in the named fluxes on a platinum wire, and the pearl treated in the reducing flame. After oxidation, the glasses appear amethystine, and by reduction colorless. The amethyst tinge is not affected on exposure to the oxidating flame, if a substance at the same time contains other coloring ingredients, but is in the reducing flame. If the presence of manganese is considerable, the pearl must be suddenly removed after reduction, because the color returns if allowed to cool gradually, owing to a higher state of oxidation ensuing.

When the quantity of manganese is so inconsiderable that it affords no amethyst color, either with borax or microcosmic salt, in the oxidating flame, a microcosmic glass must be formed, in which a sufficient quantity of the substance under examination has dissolved, and brought in contact with a crystal of nitre, as described with the reagents, page 52 ; by which means the assay despumates, and the pellicle, on cooling, assumes, in proportion to the quantity of manganese present, an amethystine or slightly rosy appearance. This procedure is not available in compound combinations, in which other metals or metallic oxides that likewise impart a color to the borax or microcosmic glass present themselves ; and recourse must be had to soda, which is decidedly the most characteristic reagent for manganese. In a substance containing less than 0.1 per cent. of oxide of manganese, the detection of this metal succeeds very readily in the following manner :—The substance under examination is reduced to an impalpable powder, mixed with from two to three times its volume of carbonate of soda, and the mixture melted in the oxidating flame on platinum foil. The oxide of manganese dissolves in the soda, forming a transparent green mass, which flows round the undissolved portion, and appears distinctly bluish-green on cooling. Should the quantity of manganese be less than 0.1 per cent., this green appearance is not produced with soda alone, but when two parts of this alkali, and one part nitre, are applied, all the manganese is converted into a higher oxide ; the soda is colored characteristically green, even by the smallest trace of this ingredient, which color becomes distinct on cooling. A similar method must be employed for the determination of manganese in Rose Quartz. The fine quartz powder, with soda and nitre, is kneaded into a paste with water, placed in the ring of the platinum wire, and melted to a pearl before the oxidating flame. This pearl is, while hot, transparent and colorless ; on cooling, opaque ; and when cold, slightly, but very distinctly, green.

If the substance consists of metallic sulphides and arsenides, it must be well roasted on charcoal, previous to the treatment on platinum wire, with soda, or soda and nitre.

When manganese forms a constituent of a substance, contain-

ing at the same time, for instance, silica and oxide of cobalt, as in picked ores, no green is obtained with soda, but a bluish-colored mass, consisting of silicate of soda and dissolved oxide of cobalt, which perfectly destroys the reaction of manganese. But if the silica is previously removed from the substance under examination, the slightest trace will again be perceptible. The manner of proceeding is as follows :—If the substance to be treated contains metallic sulphides and arsenides, it must be roasted and melted to a pearl, in proportion to the quantity of silicic acid, with an equal part of soda, and twice its volume of borax, on charcoal in the reducing flame. If arseniated metallic oxides are in the assay, they become reduced, and cohere, forming a readily fusible metallic globule. This is generally the case with cobalt or nickel ores, where arsenic-cobalt, arsenic-nickel, or the three metals, are obtained in combination. On cooling, the glass is separated from the reduced metallic arsenides, powdered, dissolved in hydrochloric acid, and perfectly evaporated to dryness ; the metallic chlorides dissolved in water, and separated from the silicic acid by filtration ; oxide of cobalt, oxide of nickel, protoxide of iron, protoxide of manganese, *et cetera*, precipitated from the solution by potassa, in an excess of which the alumina dissolves. The precipitate, separated by filtration, washed, and dried, is examined for manganese with soda and nitre, as already described.

§ 3. ZINC—Zn—*Presence in the Mineral Kingdom, and in the Scoriae of Smelting Furnaces.*

Zinc is found in nature :—

a. With Sulphur, as in *Zinc Blende* [Zn S], which is often contaminated with other Sulphides ; *exempli gratia*, Sulphides of Iron, Lead, Cadmium, Silver, *et cetera*.

b. In an oxidized state, with Earths, or other Metallic Oxides, as with Alumina, Magnesia, and Protoxide of Iron, also Aluminous Oxide of Zinc, in *Automalite* or *Gahnite*, which is an Aluminate of Zinc and of Iron. By some mineralogists it is considered as a variety of Spinel, and as it contains twenty-five to

thirty per cent. of the Oxide of Zinc, it has been called *Zinciferous Spinel*,—

= $\left[\begin{smallmatrix} \text{Zn} & \text{O} \\ \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \end{smallmatrix} \right] \text{Al}^2 \text{O}^3$; some varieties contain Silicic Acid, and traces of Manganese, Lime, Sulphur, and Cadmium; with a small quantity of Iron and Manganese, in *Red Oxide of Zinc*—usually mixed with some Red Oxide of Manganese—and with a large proportion of Iron and Manganese, in *Franklinite*, or *Zinc Oxyde Ferrifère*. The formula, according to KOBELL, is $\left[\begin{smallmatrix} \text{Mn} & \text{O} \\ \text{Fe} & \text{O} \\ \text{Zn} & \text{O} \end{smallmatrix} \right] \text{Mn}^2 \text{O}^3$. This formula, supposing the Manganese to exist in the state of Red Oxide, can be represented as follows:— $\left[\begin{smallmatrix} \text{Fe} & \text{O} \\ \text{Zn} & \text{O} \end{smallmatrix} \right] \left\{ \begin{smallmatrix} \text{Mn}^2 & \text{O}^3 \\ \text{Fe}^2 & \text{O}^3 \end{smallmatrix} \right\}$. According to GRAHAM, Red Oxide of Manganese $[\text{Mn}^2 \text{O}^3]$ is a double oxide, being a compound of single equivalents of Protoxide $[\text{Mn} \text{O}]$ and Sesquioxide of Manganese $[\text{Mn}^2 \text{O}^3]$. It forms the mineral Hausmanite, which differs from Manganite in having Protoxide of Manganese in the place of Water.

c. As an Oxide, with Sulphuric Acid and Water, in *Sulphate of Zinc* $[\text{Zn} \text{O}, \text{S} \text{O}^3 \text{H} \text{O} + 6 \text{aq}]$;

d. As a Carbonate, in *Calamine* $[\text{Zn} \text{O}, \text{C} \text{O}^2]$; and with Carbonic Acid and Water, in *Zinc Bloom* $[2 (\text{Zn} \text{O}, 3 \text{H} \text{O}) + 3 (2 \text{Zn} \text{O}, \text{C} \text{O}^2)]$; and,

e. In combination with Silicic Acid and Water, in *Silicious Oxide of Zinc* $[2 (3 \text{Zn} \text{O}, \text{Si} \text{O}^2) + 3 \text{aq}]$; Zinc is also found as an accompanying ingredient in many Silver, Lead, and Copper furnace products; as in the *Rohstein*,—the product of the first smelting of Copper Ores;—*Bleistein*,—which is a similar product, obtained in the smelting of cupriferous and other Lead Ores,—*Kupferstein*,—obtained by smelting wasted Bleistein with the slags deposited in the Rohstein process, or with Quartzose Copper Ores. It contains from twenty-eight to forty per cent. of copper, and is principally composed of the Sulphides of Copper, Lead, Iron, and Silver;—*Tutty, et cetera*. Zinc is also met with in Bleiglanz, Voltzite, Rothzinkerz, Kieselzinkerz, and Jeffersonite.

Examination for Zinc.

This examination is very simple ; the substance either containing much or little sulphide of zinc, or else this metal in an oxidized state, is treated with a sufficient quantity of carbonate of soda, upon charcoal, in the reducing flame. Metallic zinc volatilizes, but on coming in contact with the air is again oxidized. When a considerable quantity of this metal is present, the zinc flame is produced, and the charcoal becomes coated with flowers of zinc ; but if a small quantity only, the charcoal is covered with an oxide at a short distance from the assay, and no alteration is observed in the flame.

The sublimate, which when hot is yellow, and after refrigeration white, gives, when treated in the oxidating flame with a few drops of cobalt solution, a beautiful characteristic green color, which is properly recognized only on cooling. When the assay contains much lead, or if an operator is examining metallic lead for zinc, he will find, on treating the substance in the reducing flame, that the oxide of zinc sublimate is not deposited so far from the assay as the lead one, but it may often occur that the lead sublimate is mixed with zinc. To determine if such has taken place, heat the sublimate with a solution of cobalt in the oxidating flame ; the lead, if any is present, becomes reduced by the glowing charcoal, and volatilizes, while the oxide of zinc remains, and gives the characteristic green tinge. Too strong a blast must not be directed upon the moisture, or else the zinc will separate from the charcoal, and is therefore apt to be blown away. This can be avoided by rubbing the spot upon the charcoal where the sublimate generally rests, with a few drops of a cobalt solution ; a single drop of the liquid is sufficient for ascertaining the presence of a very small proportion of zinc. When this metal is present in minute quantities only, a strong flame must be deflected upon the substance, which will then become a pyrophorus, and if the assay contains lead or bismuth, it will volatilize.

§ 4: COBALT—Co—*Presence in the Mineral Kingdom, and in the Scoria of Smelting Furnaces.*

Cobalt is found in the following states :—

a. Metallic, mixed with other metals, as with Arsenic, and sometimes traces of Iron, Copper, and Sulphur, in *White Cobalt* [Co, 2 As], at times [Co, 3 As] ; with Arsenic, Iron, and sometimes a little Manganese, in *Tin-White Cobalt* [Co As + Fe As] ; with Selenium and Selenide of Lead, in Selenide of Lead and Cobalt [Co, 2 Se + 3 (Pb Se)], *et cetera* ;

Rose's analysis of the Selenide of Lead and Cobalt :—

Lead	63.92
Cobalt	3.14
Selenium	31.42
Iron	0.45

98.93

b. As a Sulphide, in *Cobalt-kies* [2 Co, 3 S], often with traces of Sulphide of Iron, and Sulphide of Copper ; and with Arsenic and a trace of Iron, in *Bright White Cobalt* [Co, 2 S + Co, 2 As] ;

c. As an Oxide, with Manganese and water, in variable proportions, in black, brown, and yellow *Earthy Cobalt* ;

d. As Oxide, with Sulphuric Acid and Water, in *Red Vitriol* [3 Co O, S O^s + 8 aq] ; WINKELBECH analyzed a compound which gave the following formula,—[Mg O, S O^s + 3 (Co O, S O^s) + 28 aq] ;

e. Also in an oxidized state, with Arsenic Acid and Water, in *Cobalt Bloom* [3 Co O, As O^s + 6 aq] , and *Nickelochre*.

This metal also occurs, in a small proportion, in different furnace products ; namely, in such as are produced from ores. To these belong, Lead and Cobalt Speiss, the principal constituents of which are Nickel and Arsenic, and perhaps also many Iron, Lead, and Kupfer-steins, which fall with the slags, *et cetera*.

Examination for Cobalt.

Cobalt is a metal which is oxidized with great facility, and in

this state it imparts to the glasses of borax and microcosmic salt a beautiful blue color, both in the oxidating and reducing flames ; but simple and complex combinations cannot be treated in this manner.

If, in the previously described minerals, selenide of lead and the selenide of lead and cobalt are excepted, this metal can be easily determined, if those which are combined with sulphur and arsenic are first roasted upon charcoal, and their fused assays treated with borax in the oxidating flame. The minerals and products containing neither manganese nor iron, give immediately a beautiful blue glass, which, if a great addition of the substance is present, appears black and opaque. If a large quantity of manganese or iron is present, the glass in the first case will be violet, and then green ; but if it is operated upon in the reducing flame, the red color of the manganese and the yellow of the iron disappear, the bead acquires a bottle-green tinge, and then becomes either pure blue from cobalt, or greenish-blue from iron and cobalt. If copper or nickel forms a part of the mineral, the oxides of these metals also dissolve in the borax, and color the flux so intensely that the characteristic cobalt blue is not perceptible. If such a glass be treated sufficiently long in the reducing flame, these oxides are deposited in the metallic state, and the blue cobalt color appears, which, if iron is at the same time present, is mixed with a bottle-green hue.

The selenide of lead and selenides of lead and cobalt, also easily fusible furnace products, in which cobalt forms only a small constituent, are treated alone ; but the difficultly fusible furnace products are mixed with two or three times their volume of proof lead, and treated with borax on charcoal in the reducing flame, until the glass partakes of the color imparted to it by the readily oxidizable and non-volatile metals. If the glass be covered with the apex of the reducing flame, so as to allow a free access of atmospheric air, only cobalt, iron, and manganese oxidize, these dissolve in the flux, and a more certain determination is effected. In the treatment of a substance containing cobalt, iron, and manganese, the iron is converted into black oxide, giving a bottle-green color, and the manganese becomes protoxide, and dissolves

without coloring the flux ; therefore, the cobalt blue and bottle-green colors only result : the green tinge is very readily distinguished from the color imparted by sesquioxide of iron, *per se* in the reducing flame, if the quantity of cobalt be very small. The presence of cobalt can also be recognized, if a portion of the glass, free from metallic particles, is fused in the ring of the platinum wire, and the assay then treated for a short time in the oxidating flame. The iron in this case becomes sesquioxide, and the glass assumes a green color, produced by the yellow from the iron and the blue from the oxide of cobalt. When an exceedingly minute quantity of cobalt is present, the glass only partakes of the ferruginous tint, which is not affected by a small quantity of manganese, but may be destroyed if there is much present. If the mineral under examination contain, besides cobalt, copper, nickel, bismuth, zinc, iron, antimony, and arsenic, a great part of the zinc, antimony, arsenic, as well as a small portion of bismuth and lead,—which is added,—will be volatilized, and after the substance is treated with borax, the copper and nickel remain behind unchanged. Should the glass, after subjection to a good reducing flame, present no cobalt or cobalto-ferruginous color, but quite a foreign aspect, the assay must again be heated, and during the fusion a small quantity of the liquid glass removed from the metallic bead with the forceps, and treated for a long time upon another part of the charcoal with the oxidating flame ; the reducible metallic oxides are precipitated, and the glass, upon refrigeration, acquires only the cobalt or cobalto-ferruginous tinge.

§ 5. *NICKEL—Ni—Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Nickel occurs in nature as—

a. Metallic, with Iron, in *Native Iron* ; with Arsenic, in *Arsenical Nickel—Kupfernickel*—[Ni, As] ; which sometimes contains traces of Cobalt, Iron, Manganese, and Sulphide of Lead ; and with a larger proportion of Arsenic, in the *Weissnickel* of the German mineralogists [Ni, 2 As] ;

b. In the state of a Sulphide, either *per se*, or combined with other Sulphides, namely, in a simple combination in *Sulphide of Nickel—Haarkies*—[Ni S]; in compound combinations with Antimony and Arsenic, in *Antimonial Nickel—Nickelspiesglanzerz*;—with Arsenic and Iron, in *Nickelglance* [Ni, 2 S + Ni, 2 As]; and as an exceedingly small constituent in *Siberian Needle Ore*;

c. As Oxide, with Arsenic Acid and Water, together with traces of Cobalt, Iron, and Sulphuric Acid, in *Nickel Ochre* [3 Ni O, As O₅ + 9 aq]. Further, Nickel also forms a constituent of many of the products of smelting works, when the ores employed contain it. Nickel either concentrates itself in Roh-, Blei-, and Cupfer-stein, in the fusion of cupriferous Silver and Lead ores, or it deposits as a peculiar product in combination with Arsenic and other Arsenious alloys, called Lead Speiss. It is also found in Schwartz-kupfer, obtained from the above mentioned ores, or as a principal constituent in combination with Arsenic, in the Cobalt-speiss, which is deposited in the preparation of Smalts. Nickel is likewise found in Speiss-cobalt, Lavendulan, Pimelite, Olivine, and Chrysolite.

Examination for Nickel.

This examination is not difficult, provided the substance under investigation does not contain a combination of different-metallic sulphides and arsenides; but far more difficult and tedious when any complex combinations occur. The above mentioned substances will be reviewed singly.

Native Iron, which contains only a very small quantity of Nickel, should be treated with borax on charcoal, in the oxidating flame, until the glass becomes of such a deep green hue that it appears almost opaque on cooling,—as long as metallic iron is present, the iron dissolved cannot be perfectly raised to the state of sesquioxide;—the undissolved iron should then be removed with the forceps from the fluid, and the latter subjected for a considerable time to the reducing flame. If the iron contains nickel, the latter will be dissolved as oxide with it, but will be again precipitated in the metallic state by a perfect reducing flame, and

may be seen with the microscope in a finely-divided state, and of an almost silver white color, on the side of the bead tinged only with protoxide of iron. If the quantity of nickel present is so small, that it cannot be recognized in the metallic state by this method, the experiment should be repeated in the following manner :—The nickeliferous iron is to be first treated on charcoal with a moderately large quantity of borax, in the oxidating flame, until the glass is saturated ; the undissolved iron is then to be removed from the fluid glass, and replaced by a small piece of pure lead. The glass must next be subjected to a good reducing flame, in order to reduce the nickel so that it may form a button with the lead, and the whole allowed to cool. During the refrigeration a part of the nickel separates and forms a pellicle, which at first moves about irregularly on the surface of the still fluid lead button, but, on the solidification of the lead, becomes fixed, and when perfectly cold appears greyish-white. If the quantity of nickel present be not extremely minute, the whole surface of the button will be coated, but if the contrary be the case, a part only ; if none be present, the surface of the lead will be bright on cooling.

The borax glass, saturated with protoxide of iron and oxide of nickel, after subjecting it, with the addition of lead, to the reducing flame, until all the oxide of nickel is reduced, can also be finely pulverized, and the powder treated with water in a porcelain capsule over the spirit-lamp. The borax glass dissolves in the water, and the metallic nickel, with the protoxide of iron, remains behind. If the solution be then cautiously poured off, and replaced by pure water, the presence of metallic nickel in the residue may be soon recognized, by dipping a magnetic bar a little below the surface of the water, and carefully observing if any metallic particles are attracted by it.

The presence of Nickel in Arsenical Nickel—Kupfernickel, Sulphide of Nickel—Haarkies, Antimonial Nickel—Nickel-spiesglanzerz, Nickelglance, and in the Lead and Cobalt Speiss, may be easily detected : it is merely necessary to heat a fragment in a glass tube, then roast it on charcoal, and treat the roasted specimen with borax in the oxidating flame, when the color of oxide of

nickel is obtained. If such a glass be subjected to the oxidating flame, the nickel will be reduced and deposited on the side of the bead. If the substance contains cobalt or iron, the glass becomes colored blue or green ; if, on the other hand, it be free from them, it will be colorless. When the roasted substance, which still generally contains arseniate of nickel, is fused with soda and borax on charcoal in the reducing flame, a white metallic button is obtained ; which is not, however ductile, but which is attracted by the magnet, and must therefore be a combination of nickel with arsenic, in very small proportion. The presence of nickel in these substances may also be recognized in the following manner :—The matter is fused on charcoal in the oxidating flame, until the greater part of the arsenic is driven off, and when antimonial nickel is under examination, the greater part of the antimony also ; the residue combines, forming slightly fuming particles. These grains are then treated for some time with borax in the oxidating flame, in order to separate the easily oxidizable metals—iron and cobalt—and the whole allowed to cool. When cold, the grain is to be separated from the glass, and again fused on charcoal with borax in the oxidating flame. If the whole of the iron and cobalt were removed by the first treatment, the glass now exhibits the color of nickel only, but if a trace of these metals remains behind, the glass is immediately colored by their presence ; in this case, the treatment of the metallic particles must be performed a third time, when the proper color of oxide of nickel is obtained. In needle ore, as well as in cupreous iron and Bleistein, and in the plumbiferous Kupferstein, the presence of a small trace of nickel may be detected in the following manner :—The substance is to be first well roasted with carbonate of soda and borax, and from 50 to 100 milligrammes of proof lead fused on charcoal in the oxidating flame. The button of lead thus obtained contains the easily reducible metals of the roasted substance, and the nickel also ; this may be recognized during the refrigeration of the button, in the way already given under the head Native Iron. Nickel ochre may be recognized as oxide of nickel, by its behavior to borax and microcosmic salt ; if the glass containing the oxide of nickel be treated on charcoal in the reducing flame, the

nickel will be separated in the metallic state, and the glass sometimes shows a slight cobalt tinge. Treated with soda and borax together, on charcoal, nickel ochre is readily reduced to an easily fusible metallic button, which, on cooling, becomes white and brittle ; it communicates the peculiar color of nickel to the fluxes in the oxidating flame.

Plombo-Nickeliferous Black Copper, which often contains a number of other metals, namely, arsenic, iron, zinc, antimony, and cobalt, when examined for nickel, must be first treated with boracic acid on charcoal, and the glass kept always covered with the reducing flame, in order that the copper and nickel may not be dissolved, while the other metals are partly volatilized, and partly taken up as oxide by the acid. When the metallic button has been so far refined that it begins to be coated with a thin incrustation, which consists of oxide of nickel, the process should be stopped, the solid copper button removed with the forceps from the fluid slag, placed on the anvil, and struck once or twice with the hammer, in order to free it perfectly from the dross. Though this button is malleable, it does not possess the color of pure copper, but is rather white, according as the quantity of nickel present is large or small. If it be desired to examine further into the presence of nickel, the button must be treated with microcosmic salt, on charcoal in the oxidating flame, and the glass observed, whether it becomes of a beautiful green color, and whether it retains this tint on cooling. This green color is derived from the combined greenish-blue of copper and the reddish-brown of nickel ; in this case the greenish-blue, resulting from a large quantity of copper, which alone, borax glass exhibits on cooling, should be observed. As nickel is more oxidizable than copper, this beautiful green color is obtained even when the quantity of nickel present is very small.

The following simple and decisive method for the detection of nickel, when contained in large quantities of cobalt, has been arrived at by the German author :—Fuse in the oxidating flame, to a bead, a moderate quantity of borax, in the loop of a platinum wire, with sufficient oxide of cobalt to give an opaque glass ; remove the assay, and prepare one or two similar beads, and place

the whole in a charcoal cavity, with a button of pure gold, weighing from 50 to 80 milligrammes. The operator must now treat in the reducing flame, until he is satisfied that the whole of the nickel is in a metallic state ; the charcoal during the action must be inclined alternately backwards and forwards, so that the gold button may flow through the molten glass, and form an alloy with the reduced particles of nickel. When the golden globule solidifies, it must be extracted with a forceps, placed between paper, and struck with a hammer so as to detach all the adhering vitreous parts. The auriferous button, which has become more or less grey, from the presence of nickel, and also more frangible than pure gold, is now to be mixed with microcosmic salt, and heated for some time in the oxidating flame. If the borax glass has not been in the first instance oversaturated with oxide of cobalt, a bead will be now obtained, which is colored only by oxide of nickel, and will therefore appear brownish-red while hot, and when cold reddish-yellow. Should portions of oxide of cobalt be also reduced, as the cobalt is oxidized before the nickel, either a blue glass, colored by oxide of cobalt, or a green one—if some nickel was also oxidized—will be obtained. In either case the glass must be separated from the button, mixed with more microcosmic salt, and heated in the oxidating flame until it acquires a tinge. If the borax glass had not been oversaturated at the commencement, the color now obtained will proceed from nickel, although the oxide of cobalt contains a trace only ; but if oxide of nickel be not present, the microcosmic bead remains perfectly colorless. The auriferous button, which still contains nickel, after treatment with microcosmic salt, can be obtained again in a pure state, by smelting the alloy on charcoal, with a quantity of pure lead, and then cupelling.

§ 6. *IRON—Fe—Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Iron occurs very abundantly in nature, thus :—

a. Metallic, as *Native Iron*, which contains some per centage of Nickel ;

b. In a state of Sulphide, as well *per se* as in combination with

other Sulphides and Arsenides of metals; namely, *per se* in *Magnetic Iron Pyrites* $[\text{Fe}, 2 \text{S} + 6 (\text{Fe}, \text{S})]$; in *Common Pyrites* $[\text{Fe}, 2 \text{S}]$; with Arsenical Iron, in *Arsenical Iron* $[\overset{\text{Fe}}{\text{Co}}\} 2 \text{S} + \{\overset{\text{Fe}}{\text{Co}}\} 2 \text{As}]$; with Sulphide of Copper, in *Copper Pyrites* $[\text{Cu S} + 2 \text{Fe S}]$; and *Purple Copper* $[3 \text{Cu S} + \text{Fe}^2 \text{S}^3]$; with Sulphide of Antimony, in *Hardingerite* $[3 \text{Fe S} + 2 \text{Sb}^2 \text{S}^3]$; with Sulphide of Antimony, Sulphide of Silver, Sulphide of Copper, and Sulphide of Zinc, in *Weisgiltigerz* and *Graugiltigerz*; with the same Sulphides and an admixture of Sulphide of Arsenic, in *Grey Copper* $[\overset{4 \text{Fe S}}{\text{Zn S}}\} \{\overset{\text{Sb}^2 \text{S}^3}{\text{As}^2 \text{S}^3}\} + 2 (4 \text{Cu}, \text{S}) \{\overset{\text{Sb}^2 \text{S}^3}{\text{As}^2 \text{S}^3}\}]$; with Sulphide of Copper and Sulphide of Arsenic, in *Tennantite*; and with Sulphide of Copper, Sulphide of Arsenic, a little Sulphide of Silver, and Sulphide of Antimony, in the *Copper Blende*;

c. As Protoxide or Sesquioxide, as well *per se* as combined with Metallic Oxides and Water; namely, as pure Oxide, in *Specular Iron* $[\text{Fe}^2 \text{O}^3]$ —*Red Ironstone*, *Bloodstone*;—as Oxide, combined with Water, and often with small quantities of Oxide of Manganese and Silicic Acid, in *Brown Iron Ore* and *Bog Iron Ore* $[2 \text{Fe}^2 \text{O}^3 + 3 \text{H O}]$, which last contains some Silicic and Phosphoric Acids; as Oxide, contaminated with earthy substances, in *Argillaceous Iron Stone*—*Thoneisenstein*;—as Black Oxide, with a very small admixture of Magnesia, in *Oxidulated Iron Ore* $[\text{Fe O} + \text{Fe}^2 \text{O}^3]$; as Oxide, with Oxide of Chromium, Alumina, and Magnesia, in *Chromate of Iron* $[\overset{\text{Fe}^2 \text{O}^3}{\text{Mg O}}\} + \{\overset{\text{Cr}^2 \text{O}^3}{\text{Al}^2 \text{O}^3}\}]$; as Sesquioxide, with Oxide of Zinc, and Protoxide of Manganese, in *Franklinite* or *Dodecahedral Iron Ore*; and as Protoxide, with Oxide of Zinc, Alumina, Magnesia, and a very little Silicic Acid, in *Automolite*;

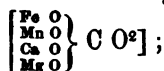
d. As Protoxide, with Titanic Acid, in various degrees of saturation in *Titaniferous Oxidulated Iron*, *Crichtonite*, *Menaccanite*, *Nigrin*, *Iserine*, *Ilmenite*, *et cetera*;

e. As Protoxide, with Tungstic Acid and Tungstate of Protoxide of Manganese, in *Wolfram*;

f. As Sesquioxide, with Arsenic Acid and Water, in *Skorodite*; as Black Oxide, with Arsenic Acid and Water, in *Arseniate of*

Iron—Cube Ore—[3 Fe O , $\text{As O}^5 + 3 \text{ Fe}^2 \text{ O}^3$, $2 \text{ As O}^5 + 18 \text{ aq}$]; as Sesquioxide, with Arsenic Acid and Water, in *Pitchy Iron Ore* [$2 \text{ Fe}^2 \text{ O}^3$, $\text{As O}^5 + 12 \text{ H O}$];

g. As Protoxide, with Carbonic Acid, and often with Carbonate of Protoxide of Manganese, with traces of Lime, Magnesia, and Water, in *Spathose Iron*, the general formula of which may be expressed by the following symbol—



h. As Protoxide, with Sulphuric Acid and Water, in *Native Green Vitriol* [Fe O , $\text{S O}^3 + 6 \text{ aq}$]; as Oxide, with Sulphuric Acid and Water, in *Vitriol-oker* [$2 \text{ Fe}^2 \text{ O}^3 \text{ S O}^3 + 6 \text{ aq}$];

i. As Protoxide, with Phosphoric Acid and Water, in *Phosphate of Iron* [8 Fe O , $3 \text{ P O}^5 + 16 \text{ aq}$] or [3 Fe O , $\text{P O}^5 + 6 \text{ aq}$]; as Protoxide, with Phosphoric Acid, and Phosphate of Protoxide of Manganese, with an admixture of Phosphate of Lime, in *Triphlite*; as Oxide, with Phosphoric Acid and Water, in *Grüneisenstein*; and as Oxide, with Phosphoric Acid, Phosphate of Alumina, small quantities of Silicic and Hydrofluoric Acids, Lime, and Water, in *Kakozene*;

k. In the oxidized state, with Silicic and Silicious combinations; namely, as Protoxide, in *Hisingerite*, *Sideroschisolite*, and *Chlorophaeite*;—BERZELIUS considers these three Silicates as simple, in which the intermingled substances are to be looked upon as extraneous;—as Protoxide, with Silicate of Protoxide of Manganese, Protochloride of Iron and Water, in *Pyrosmalite*; as Protoxide, with Water and a little Alumina, in *Chamoisite*; as Protoxide, with Lime, Protoxide of Manganese, and Sesquioxide of Iron, in *Lievereite* [$2 \left\{ \frac{3}{2} \text{ Ca O}, \frac{81}{81} \text{ O}^3 \right\} + 2 \text{ Fe}^2 \text{ O}^3$, Si O^2]; as Protoxide, with Soda, Water, and a little Lime, in *Krokydolite*; as Sesquioxide, with Water, and a little Protoxide of Manganese and Alumina, in *Chloropal*; as Sesquioxide or Protoxide, with Protoxide of Manganese, and generally with a little Alumina, Lime, and Magnesia, in many *Garnets*; as Sesquioxide, with a little Peroxide of Manganese, and Lime, in *Achmite*; as Protoxide, with Magnesia, and sometimes with the Oxides of Nickel and Manganese, in *Chrysolite* and *Olivine*; as Protoxide, with Protoxide of Cerium and

Yttria, in *Gadolinite* ; and as a secondary constituent in a number of other Silicates. Iron is met with in Kyrosite, Sternbergite, Cronstedtite, Humboldtite, Arsenioderite, Wurfelerz, Symplectite, Tantalite, Thuringite, Fayalite, Stilpnomelan, Pinguite, Knebelite, Nontronite, Anthosiderite, Eisengranate, Rhodalite, Triphylite, Tetraphylite, Vivianite, Delvauxite, Eisenapatite, Hetepozite, Huraulite and Fibroferrite.

It also occurs in many furnace products, from Silver, Lead, Copper, and Tin ores, and in most slags, either as a principal or secondary ingredient. It would be superfluous to allude further to these products, as a part of the iron contained in the ores to be smelted, and the raw products which are to undergo further operations, is generally so long retained by them, that its presence can be detected even in Black Copper and Tin.

Examination for Iron.

The test for Iron is, that the sesquioxide, as well as the protoxide, communicates a peculiar color to borax and microcosmic salt, and that the iron is with difficulty separated from both these fluxes in a metallic state only. The operator should consider whether he has to do with metallic alloys alone, or combinations of metal with arsenic, or with oxides. If the substance under examination be of pure metallic alloys, composed of difficultly fusible metals, it should be fused with borax on charcoal, in the oxidizing flame, until the operator is certain that, besides other metallic oxides, protoxide or sesquioxide of iron has been dissolved also. If the compounds, however, contain much lead, tin, bismuth, antimony, or zinc, and readily fuse, the reducing flame should be employed ; the flame should be principally directed on the glass, in order that too much of the latter metals may not be oxidized and dissolved. In both cases the glass, while still soft, is either removed from the metallic particle, and treated on any other part of the charcoal, in the reducing flame,—where the easily reducible metals will be separated, and the borax glass appear colored of a bottle-green, from the black oxide of iron, provided only oxide of cobalt prevents the reaction,—or it is immediately treated, together with the particles of alloy, in a pure

strong reducing flame, when the reducible metals which may happen to be dissolved are also precipitated in the metallic state, and again combine with the remainder of the alloy. If the alloy contain much tin, or if the bottle-green glass be treated on another part of the charcoal, with a particle of tin, for a few moments, in the reducing flame, the iron will be perfectly reduced to the state of protoxide, and the glass, on cooling, will be of a pure vitriol-green color.

If the borax exhibits a blue color, instead of the proper vitriol-green of protoxide of iron, oxide of cobalt is present, which destroys the iron tint. In this case the glass must be again softened in the reducing flame, the greater part of it removed from the charcoal with the forceps, but without taking any of the metal with it, and fused in the loop of the platinum wire. If the color is now so deep that the glass is almost opaque, it must be flattened with the forceps while still soft, a part of it struck on the anvil, and the portion still adhering diluted with more borax. The first thing then to be done, is to fuse the glass in the oxidating flame, until the protoxide of iron present is converted into the sesquioxide, and the glass is colored, while warm, of a brownish-red or yellow. If the glass contains only a slight trace of sesquioxide of iron, it appears green while hot, and pure blue when cold. If the quantity of iron be somewhat considerable, the glass will be dark green while hot, and when cold, of a beautiful green, because sesquioxide of iron, when not in excess, communicates to borax glass a yellow color when cold.

The combination of metals with sulphur and arsenic can be examined for iron in two ways. According to the first method, the specimen should be roasted on charcoal; small portions are then to be gradually dissolved in borax, on the platinum wire in the oxidating flame, and the glass examined while hot and when cold, in order to see if it displays any color. The proper iron tint is obtained immediately, with many of these compounds, which contain only metals whose oxides do not produce very intense colors; with many others, it is not however obtained, as, for example, those which contain copper, but on the contrary a green color results, which becomes lighter during the refrigeration,

and when cold, possesses a color composed of the yellow of oxide of iron, and the blue of oxide of copper. In this case the glass must be shaken from the platinum wire—see page 67,—and treated on charcoal in the reducing flame until the copper separates, and the glass exhibits the peculiar bottle-green tint of black oxide of iron, if oxide of cobalt is not present at the same time. The bottle-green glass can also be treated with tin, in order to recognise the vitriol-green color of iron. The second method is as follows :—The pulverized substance is mixed with proof lead and borax, and the whole fused on charcoal in the reducing flame, until the borax glass is colored by the difficultly reducible metals present. In the commencement, the mixture should be covered with the reducing flame ; but as soon as the borax has run to a globule, the flame should be directed only on the latter, and free access of air allowed to the fused metal.

Compounds which readily fuse on charcoal, *per se*, can be treated without proof lead. An exceedingly small quantity of iron may in this way be detected in many Lead-Glances, particularly when the glass is treated with tin. If the borax exhibits a blue instead of a green color, the glass must be further treated in the manner mentioned above for the metallic compounds.

In the compounds of oxide of iron with other metals, or with earths and acids, the iron is also best detected by fusion with borax.

Such compounds as may be supposed to contain neither oxides of copper, nickel, chromium, nor uranium, should be dissolved with borax on the platinum wire, in the oxidating flame, and the colored bead examined while hot, and also during the refrigeration ; if it exhibits the color of iron, or of iron and cobalt together, which has been already noticed, the examination is complete ; but if it presents another color, as violet passing into red, the glass must be treated for some time longer in the reducing flame, by which the violet which is derived from manganese disappears, and the bottle-green color of black oxide of iron remains. If such a substance contains much manganese, the glass treated in the oxidating flame will be a perfect dark red while hot, and red when cold. In this case it is not possible to convert the

manganese into protoxide on the platinum wire, it should therefore be removed from the wire to charcoal, and treated with tin, when the color of the manganese disappears, and the vitriol-green color of the protoxide alone remains, if oxide of cobalt is not present.

If a substance contains cobalt also, besides iron and oxide of manganese, the glass treated with the oxidating flame appears more or less of a dark violet color, which becomes, when held for a short time in the reducing flame, green while hot, and blue when cold; for example, this is the case with brown earthy cobalt, which really consists of oxide of cobalt and hydrate of oxide of manganese, but which is contaminated with oxide of iron. In a combination of much oxide of manganese, and oxide of cobalt, with very little oxide of iron, the last may be readily detected by fusing the substance with bisulphate of potassa, dissolving the resulting mass in water, adding a few spoonfuls of chloride of ammonium to the solution, warming the whole, and, when the salt is liquefied, precipitating the oxide of iron by ammonia. If the iron exists in the substance as protoxide, a few drops of nitric acid should be added to the sulphuric acid solution, and the whole warmed, in order to convert the protoxide into the sesquioxide. The oxide of iron can then be filtered from the solution, edulcorated, and tested with borax on the platinum wire. If the mineral under examination contains oxide of copper or nickel, it is always better to dissolve it with borax on charcoal, in the oxidating flame, then treat it in the reducing flame, in order to separate the copper and nickel in the metallic state, and thus obtain the proper ferruginous tint alone. If a blue glass is obtained, it must be re-oxidized on the platinum wire, in the way already mentioned. When such a substance contains oxide of chromium, a green glass is obtained with borax, which shows the presence of chromium only. In such cases, the substance must either be mixed with three parts of saltpetre and one of borax, this mixture gradually fused in the loop of the platinum wire, the chromate of potassa thus formed dissolved in water, and the residue, after edulcoration with water, dissolved with borax on a platinum wire, when the

proper color of iron will be obtained, provided no other colored oxide be present, and all the oxide of chromium removed ; or the iron can be reduced by carbonate of soda on charcoal, and the metal obtained by rubbing the glass in a mortar, and separating the nonmetallic parts. If the substance contains uranium, the ferruginous color will also be obtained, but it will not proceed from the iron alone, but partly from uranium, which yields almost the same color as iron. To obtain the proper color of iron, the substance should be melted with bisulphate of potassa, the fused mass dissolved in water, the protoxides of iron and uranium present converted into sesquioxides, by heating the solution with a few drops of nitric acid, and an excess of carbonate of ammonia added. The oxide of uranium, which is at first precipitated along with the oxide of iron, redissolves, so that the latter can be obtained by filtration, and, after washing well with water, may be tested with borax. When the ammoniacal solution is boiled, the oxide of uranium falls as a yellow powder, and can also be tested before the Blowpipe, being easily recognized with microcosmic salt.

If the substance contains tungstic or titanitic acids, the proper color of oxide of iron is obtained with borax and microcosmic salt, in the oxidating flame, as both these acids yield only a slight yellow color ; in the reducing flame, on the other hand, the microcosmic salt glass becomes, during the refrigeration, more or less of a blood-red color.

The following method for distinguishing protoxide of iron from the sesquioxide, is given by CHAPMAN, in the Chemical Gazette :—

A very minute quantity of oxide of copper is to be dissolved in a bead of borax on the platinum wire until the glass acquires a slight coloration ; the substance under examination now being added to it, the whole is subjected, for an instant only, to the reducing flame, when if protoxide of iron was present in the assay-matter, the oxide of copper will be reduced to suboxide, forming small red spots or streaks which become visible as the glass cools. The oxide of iron is converted into sesquioxide at the expense of the oxygen of the copper.

In the above experiment, if the glass were exposed for too long

a time, the oxide of copper might be reduced even if the substance under examination contained only sesquioxide of iron, as this would be converted by the flame into protoxide, and thus act, as before stated, on the oxide of copper ; and if, furthermore, this latter substance were contained in too large a quantity in the borax glass, it might become reduced by the sole action of the flame, and thus give rise to an error. To obviate, therefore, all doubt as to the presence or absence of protoxide of iron, the same authority proposes that the operation should be conducted in a different manner, which gives certain results.

The borax bead must be colored by a sufficient quantity of oxide of copper to render it of a fine blue color, but transparent when cold. To this the substance under examination in powder must be added, and the bead exposed for a moment, or until the iron compound begins to dissolve, in the oxidating flame. If sesquioxide of iron alone be present, the glass will remain transparent, and of a green or bluish-green color ; but if, on the contrary, the iron is in the state of protoxide, the glass on cooling will be marked with opaque-red patches, due to the reduction of oxide of copper into suboxide, as before explained. Care must be taken not to continue the blast too long, otherwise the suboxide of copper might again be oxidized, and the whole of the protoxide of iron converted into sesquioxide. After one or two trials, however, no error can possibly arise. The reactions are not prevented by the presence of silicic or other acids.

§ 7. *CADMIUM—Cd—Presence in the Mineral Kingdom.*

Cadmium occurs only in Zinc Ores, thus :—

- a. As Sulphide, in many *Zinc-blendes* ; for example, in Greenockite, and in the *Splendent fibrous Blende*, from Przibram, and,
- b. As Oxide, in combination with Carbonic Acid, in *Silicious Oxide of Zinc* or *Galmey*.

The quantity present does not, however, exceed from one to four per cent., either in the Blendes or in the Galmey.

Examination for Cadmium.

This metal can only be recognized as oxide before the Blow-

pipe, owing to its volatility. The cadmiferous mineral should be heated for some time in a pulverized state, on charcoal, in the reducing flame, by which the metallic cadmium is volatilized, and, in contact with the air, is immediately re-oxidized. The greatest part of this oxide deposits on the charcoal, and, when perfectly cold, may be recognized by its reddish-brown color; in a thin crust, however, it appears only yellow. When the quantity of cadmium present is not more than one per cent., it is generally difficult to obtain a sublimate of oxide of cadmium in this way; but if the powdered mineral be mixed with carbonate of soda, and this mixture treated for a few moments on charcoal, in the reducing flame, a very evident sublimate of oxide of cadmium is obtained. If the blast be continued too long, a part of the zinc, will also be volatilized, and will likewise deposit as oxide on the charcoal. The oxide of cadmium is, however, always deposited further from the assay than the zinc, and may be very easily distinguished.

§ 8. *LEAD—Pb—Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Lead is pretty generally distributed in nature. It is found:—

a. Metallic, in combination with other metals, *exempli gratia*, with Tellurium, in *Black Tellurium—Pyramidal Tellurium Glance*—[Pb Te] mechanically mixed with Sulphides of Lead and Gold; with Tellurium, Gold, and Silver, in *Yellow Tellurium* [Ag Te + 2 (Pb, Te) + 3 (2 Au, 3 Te)]; with Selenium, in *Selenide of Lead*, which often contains a trace of Cobalt; with Selenium and Copper, in *Selenide of Lead and Copper* [Pb Se + Cu Se], and *Selenide of Copper and Lead* [2 (Pb Se) + Cu Se]; and with Selenium and Mercury, in *Selenide of Lead and Mercury* [Pb Se mixed with Hg Se];

b. As Sulphide alone, and in combination with other metallic sulphides, namely: *per se* in *Lead Glance* [Pb S]; with Antimony and traces of Iron, Zinc, and Copper, in *Jamesonite—Axotomous Antimony Glance*—[2 (Pb S, Sb 3 S) + Pb S]; with Antimony and Copper, in *Bournonite* [3 (Cu S) + Sb 3 S + 2

($3 \text{ Pb } 3 \text{ S} + \text{Sb } 3 \text{ S}$)] ; with Bismuth, and a little Iron and Copper, in *Plumbo-Cupriferos Sulphide of Bismuth* ; with Antimony and a small quantity of Copper, in *Zinkenite* [$\text{Pb S} + \text{Sb } 3 \text{ S}$] ; and with Bismuth, Copper, and a very small quantity of Tellurium and Nickel, in *Siberian Needle Ore* [$\text{Cu S, Bi S} + 2 (\text{Pb S, Bi S})$] ;

c. In combination with Chlorine ; as in *Chloride of Lead—Cotunnite*, from Vesuvius—[Pb Cl] ; and the *Basic Chloride of Lead* [$\text{Pb Cl} + 2 \text{ Pb O}$], from the Mendip hills of Somersetshire ;

d. In an oxidized state, with Alumina and Water, in *Plomb-gomme—Hydrous Aluminate of Lead*—[$\text{Pb O, } 2 \text{ Al}^2 \text{ O}^3 + 6 \text{ aq}$] ; and,

e. As an Oxide, with Acids, partly alone, and partly combined with other Metallic Salts, *exempli gratia*,

¹ With Carbonic Acid, in *White Lead Ore—Weissbleierz*—[Pb O, C O^2] ; and in *Black Lead Ore—Schwarzbleierz*,—of the same composition, only containing a small proportion of free Carbon ; also in *Earthy Carbonate of Lead—Bleierde*,—which is mixed with Alumina, Silica, and Sesquioxide of Iron ;

² With Sulphuric Acid, and about two per cent. of Water, in *Native Sulphate of Lead* [Pb O, S O^3] ;

³ With Arsenic or Phosphoric Acid, and Chloride of Lead, in *Brown and Green Arsenical Lead Ores* [$\text{Pb Cl} + 3 (3 \text{ Pb O, As O}^3)$] ;

⁴ With Chromic Acid, in *Prismatic Lead Spar—Chromate of Lead*—[Pb O, Cr O^3] ; with Chromic Acid and Chromate of Copper, in *Vauquelinite—Hemi-Prismatic Olive Malachite*—[$3 \text{ Cu O, } 2 \text{ Cr O}^3 + 2 (3 \text{ Pb O, } 2 \text{ Cr O}^3)$] ;

⁵ With Molybdic Acid, in *Molybdate of Lead—Gelbbleierz*—[Pb O, Mo O^3] ;

⁶ With Tungstic Acid, in *Scheelite—Tungstate of Lead*—[Pb O, W O^3] ; and

⁷ With Vanadic Acid, in *Vanadate of Lead—Vanadiniferous Lead Spar*—[$2 \text{ Pb O} + \text{Pb Cl} + 3 \text{ Pb O, } 2 \text{ V O}^3$] ;

Further, Lead forms an essential, and also an accompanying constituent in many furnace products :—

a. Metallic, combined with other Metals ; *exempli gratia*,

with Silver, in *Workable Lead*; with Copper, as *Plombiferous Black Copper*—*Bleisches Schwarzkupfer*,—*et cetera*.

b. In the state of Sulphide, combined with other Metallic Sulphides, as for example: with Sulphide of Iron, in *Bleistein*; with Sulphide of Copper, in *Bleischen Kupferstein*; and, according to the nature of the ores smelted, mixed also with various other Metallic Sulphides, in *Tutty*, *et cetera*. Lead is likewise found in Kilbrickenite, Geokronite, Boulangerite, Federerz, Bleischimmer, Plagionite, Kobellite, Weissgiltigerz, Bleihornerz, Bleiglätte, Schwerbleierz, Nussierite, Melanochroite, and Werkblei.

c. In an oxidized state, to which belongs *Glätte*—*Lithurge*—and the *Abstrich*, a greyish froth which is raked off the surface of the *Workable Lead*, in the process of extracting Silver therefrom. The Lead is first melted at a low heat, when the *Abstrich*, which is composed for the most part of Sulphides of Lead, Antimony, *et cetera*, separates. Further, the *Cupel Grounds* saturated with Oxide of Lead, obtained in the refinement of Lead for Silver, which fall in the melting of *Plombiferous Schlichs*,—metallic slimes obtained in the washing of powdered ores.

Examination for Lead.

The qualitative examination for Lead is very readily performed, in the following manner:

When plombiferous compounds, which are met with in nature and furnace products, are treated on charcoal, in the oxidating flame, they give a sublimate which is very easily recognized. Other easily volatilized metals, which may be in combination with the lead, either fume away entirely, or else deposit an oxide upon the support. The oxide of lead sublimate, which is dark lemon-yellow while hot, and sulphur-yellow when cold, deposits nearer to the assay than the sublimates of some other metallic oxides, namely, those of tellurium, selenium, antimony, and arsenic, and is by this means distinguished. Should zinc also be an ingredient, the sublimate of oxide of lead will probably be contaminated with a quantity of the oxide of this metal, but the sulphur-yellow color of the lead deposit cannot, however, be mis-

taken, when the assay has perfectly cooled. Plumbiferous sulphur compounds can be examined for lead by two methods. Either by treating them in the reducing flame on charcoal, with a small addition of borax to separate the iron, and obtain a lead sublimate, or by roasting the sample moderately on charcoal, and reducing with soda, when the lead is obtained pure by sifting the assay from carbonaceous matters, *et cetera*. If the substance contains bismuth, a deposit of this oxide will ensue in the first treatment, but it is darker than the oxide of lead; and in the second a friable metal is obtained, which, when the bismuth is present in an appreciable quantity, is supposed not to be lead. Should this occur, the substance must be smelted with bisulphate of potassa, as given under the head of Bismuth. The sulphate of lead procured in this manipulation is heated with soda on charcoal, by which means the pure metal, and an oxide of lead sublimate is obtained. If much copper had also been present, black copper would result in the second method, but being so dissimilar from the oxide of lead, no mistake could occur in the discrimination.

Combinations of lead with chlorine, and substances which contain the lead in an oxidized state, with other metallic oxides, earths, or acids, must always be treated with soda on charcoal, in the reducing flame. By this means, metallic and oxide of lead are produced, which may be procured on sifting the pyrognostic residue from scorix. Should impure lead be obtained by this method, there must have been other metallic oxides present in the specimen; therefore, to insure an infallible test of the presence of this metal, the assay must be treated for a long time in the oxidating flame, when the characteristic lead sublimate is procured.

§ 9. *BISMUTH—Bi—Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Bismuth is found in nature :—

a. Metallic, as *Native Bismuth* [Bi], which generally contains a very small quantity of Arsenic; with Tellurium, Sulphur, and

a trace of Selenium, in *Telluric Bismuth* $[\text{Bi S} + (\text{Bi, Te})]$; and also as a small ingredient in a compound of Arsenic, Cobalt, Iron, and a little Copper, Nickel, and Sulphur, with a trace of Manganese, in *Bismuth Cobalt Ore*, from Schneeberg;

b. In the state of Sulphide, *per se*, and also in combination with other Metallic Sulphides, namely: *per se*, in *Bismuth Glance* $[\text{Bi S}]$; with Copper, in *Cupreous Bismuth—Cupriferous Sulphide of Bismuth*— $[3 (\text{Cu S}) + 2 \text{Bi, } 3 \text{S}]$; with Lead, Copper, a little Nickel and Tellurium, in *Siberian Needle Ore*; and with Lead, Silver, a little Iron and Copper, in *Bismuthic Silver*;

c. As an Oxide in *Bismuth Ochre* $[\text{Bi O}^2]$; with, probably, traces of Iron and Copper; and also as an Oxide, with Carbonic Acid and Water, mixed with a little Sesquioxide of Iron, Alumina, and Silica, in *Carbonate of Bismuth*.

This metal sometimes presents itself as a secondary ingredient in many furnace products. It is found, for example, more or less in the metallic state, in many *Cobalt Speisses*, if the Cobalt Ores employed for the preparation of Smalt are not free from Bismuth. Smalt—Silicate of Cobalt—is prepared in a pure state by precipitating Sulphate of Cobalt with Silicate of Potassa. It is manufactured in very large quantities in Saxony.

Examination for Bismuth.

The simplest method for detecting Bismuth in the above-mentioned minerals and Cobalt-Speiss, is by treating those substances which contain bismuth as a metal, with or without sulphur, combined with a small portion of borax, on charcoal in the oxidating or reducing flame; and those which contain this metal in an oxidized state, treated with soda, likewise on charcoal in the reducing flame. The bismuth, which is either already met with in a metallic state, or is reduced by soda, sublimes by degrees, and coats the charcoal with an oxide, which, while hot, appears dark orange-yellow, and on perfectly cooling, citron-yellow.

If the substance at the same time contains much lead, the color of the deposit is lighter, and very similar to the lead sub-

limite ; this occurs with the Siberian Needle Ore. In such cases, the presence of bismuth cannot be ascertained with certainty by the previous method, and it is necessary to recur to other methods of procedure.

The first is :—The substance is roasted,—if it contains sulphur or arsenic, it is preferable to perform this experiment in a glass tube, to drive these off as much as possible, because it sinters readily on charcoal,—dissolved in microcosmic salt by the aid of the oxidating flame, and the glass treated with tin for a short time in the reducing flame. If the quantity of bismuth is so small that it contains less than a fourth part of the lead present in the substance, the microcosmic salt will be colored, on perfectly cooling, dark grey, and become ultimately opaque. As oxide of antimony produces the same reaction with tin in microcosmic salt, it is necessary that the absence of antimony be ascertained by a preliminary examination of this metal. If the substance, besides bismuth, also contains copper, the microcosmic salt becomes on cooling brownish-grey, nearly black, and opaque.

During the roasting of a substance, very rich with bismuth, in a glass tube, a yellowish-white sublimate is generally formed close to the assay, and even on the undermost part of the tube which melts in a strong flame to brownish orbicules ; which, on cooling are transparent, and of a yellow color. This deposit consists of oxide of bismuth.

The second method is the following, if the bismuth present is exceedingly small, and the lead not distinct :—The substance is well roasted in a powdered state, the heated mass fused in a platinum spoon, with bisulphate of potassa, and the residual matter treated with water in a porcelain basin, over the flame of the lamp, till solution takes place. By this means the sulphate of potassa and other soluble sulphates are dissolved, while basic sulphates of lead and bismuth remain. The supernatant liquor is decanted cautiously, the residue again treated with distilled water, a few drops of nitric acid added, and the whole heated. The sulphate of bismuth dissolves, while the sulphate of lead remains behind. If both salts be then separated by filtration, and the oxide of bismuth precipitated from the solution by micro-

cosmic salt, with the application of heat, a white precipitate is obtained, which dissolves either colorless or yellowish, in micro-cosmic salt : but the glass, when treated with tin in the reducing flame, on charcoal, assumes a dark greyish color on cooling, and behaves precisely like oxide of bismuth.

The artificially prepared metallic nickel, on a large scale, is not always free from bismuth ; should an operator, therefore, wish to determine a small admixture of bismuth in the nickel, before the Blowpipe, he must mix one part of the finely divided sample with two parts of saltpetre, and treat on a platinum wire in the oxidating flame. The pyrognostic assay, which consists of potassa, oxide of nickel, and oxide of bismuth, is detached from the wire, and treated for some time on charcoal in the reducing flame. The oxide of bismuth is very readily reduced by this means ; the metallic bismuth sublimes, and coats the charcoal with an oxide.

§ 10. URANIUM—U—*Presence in the Mineral Kingdom.*

Uranium, which is an exceedingly rare metal, occurs in nature in an oxidized state :—

a. As friable and compact Hydrate of the Sesquioxide of Uranium, the last of which is mixed with Lime and Oxide of Lead ; the two varieties are called *Uran Ochre* ;

b. As an Oxide, with Phosphoric Acid, and Phosphate of Lime, in *Uranite* [$3 \text{ Ca O}, \text{P O}^5 + 2 (\text{U}^2 \text{ O}^3, \text{P O}^5) + 24 \text{ aq}$] mixed with [$3 \text{ Ba O}, \text{P O}^5$] ; in an oxidized state, with Phosphoric Acid, and Phosphate of Copper, in *Chalkolite*—*Green Uranite*— $[3 \text{ Cu O}, \text{P O}^5 + 2 (\text{U}^2 \text{ O}^3, \text{P O}^5) + 24 \text{ aq}]$;

c. As Protoxide, with Titanic Acid, Lime, Oxide of Cerium, Protoxide of Manganese, Sesquioxide of Iron, Oxide of Tin, Water, and traces of Hydrofluoric Acid and Magnesia, in *Pyrochlore*, from Fredrikswärn—WÖHLER's analysis ;

d. As Sesquioxide, with Tantalic Acid and Yttria, in *Yellow Ytthro-Tantalite* [$3 \text{ Y O} \left\{ \begin{smallmatrix} \text{Ta} \text{ O}^3 \\ \text{U}^2 \text{ O}^3 \end{smallmatrix} \right\}]$; and,

e. As Protoxide, with Silicic Acid, and traces of Sesquioxide of Iron, Sulphide of Lead, and Oxide of Cobalt, in *Pitch-Blende*—*Uran-Pecherz*— $[3 \text{ U O}, 2 \text{ Si O}^2]$. Uranium is also found in

Pechuran, Uranvitriol, Uranbloom, Uranotantalum, Fergusonite, Euxenite, Polykras, and Thorite.

Examination for Uranium.

In the previously described minerals, with the exception of Chalkolite and Pyrochlore, uranium is determined by treating them in a powdered state with microcosmic salt, upon platinum wire, as well in the oxidating as in the reducing flame.—See pages 92, 93, Table II.

Pyrochlore, on account of its containing a considerable quantity of titanio acid and iron, destroys the colors given by uranium in the reducing flame; the glass, upon cooling, becomes blood-red. In the oxidating flame, however, the microcosmic salt bead becomes greenish upon refrigeration, but is contaminated with yellow. When the substance contains only a small quantity of uranium, and much iron, the ferruginous reaction is given both with borax and microcosmic salt; therefore, the sample must be fused with bisulphate of potassa, the residue dissolved in water, nitric acid added to convert the iron and uranium into sesquioxides, and then carbonate of ammonia poured into the liquid for their separation,—which is treated of under Iron.

If the mineral contains oxide of copper, as is the case with the Chalkolite, from Cornwall, a green glass is also obtained with borax and microcosmic salt, in the oxidating flame. As minerals containing proto- and sesquioxide of iron, and oxide of copper, without uranium, give a similar reaction to the above substances, when examined for this metal, they must be submitted to a different treatment. The substance is to be smelted with carbonate of soda and borax, with an addition of lead, upon charcoal in the reducing flame, until the whole of the copper is reduced and alloyed with the lead.

The glass, when cold, must be pulverized, treated with hydrochloric acid and water, the protoxides of uranium and iron, if present, converted into sesquioxides, by the addition of a few drops of nitric acid, and then carbonate of ammonia added in excess, and the subsequent part of the process conducted in the same manner as mentioned under the head of Iron. According to

LIEBIG, Uranium may be extracted from Pitch-blende—a variety of Uran Ochre—by the following process :—After heating the mineral to redness, and reducing it to an impalpable powder, it is digested in pure nitric acid, diluted with four parts of water, taking the precaution to employ a larger quantity of the mineral than the acid added can dissolve. By this process, the protoxide of uranium is converted into sesquioxide, which unites with the nitric acid, almost to the total exclusion of the iron. A current of sulphide of hydrogen gas is then transmitted through the menstruum, in order to separate lead and copper, the sulphides of which are always present in Pitch-blende. The solution is boiled to expel any free acid, and after being concentrated by evaporation, is allowed to repose. The nitrate of sesquioxide of uranium crystallizes out in flattened four-sided prisms, of a beautiful lemon-yellow color.

The sesquioxide of uranium is employed in the arts, for imparting a fine orange color to porcelain, *et cetera*.

§ 11. COPPER—Cu—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

This metal is found very abundantly in nature :—

- a. Metallic, as *Native Copper* [Cu] ;
- b. With Selenium, as well *per se*, as with other Selenides namely, *per se*, in *Selenide of Copper* [2 Cu Se] ; with Lead, in *Selenide of Copper and Lead*, and *Selenide of Lead and Copper* ; and with Silver, *et cetera*, in *Eukairite*—from the Greek, signifying *opportune* ; in allusion to its discovery just as BERZELIUS had completed his examination of Selenium—[2 Cu Se + Ag Se] ;
- c. In the state of Sulphide, alone, and in combination with other metallic sulphides, namely, *per se*, as *Vitreous Copper*—*Kupfer-glanz*—[Cu S], which generally contains traces of Iron and Lead ; with Iron, in *Purple Copper*—*Bunt-kupfererz*—and *Copper Pyrites* ; with Arsenic and Iron, in *Tennantite* [$\begin{smallmatrix} 4\text{ Fe } 8 \\ 4\text{ Cu } 8 \end{smallmatrix}$] 2 As, 3 S + 2 (4 Cu, 4 S, 2 As, 3 S] ; with Silver and a little Iron, in *Argentiferous Copper Glance*—*Silberkupfer-glanz*—[Cu S + Ag S] ; with Arsenic, Iron, a little Antimony and Silver, in

Copper Blendes ; with Tin, and a little Iron, in *Tin Pyrites* [$\text{Cu S} + \text{Sn S}$] ;—the best formula for *Tin Pyrites* $\left[\begin{smallmatrix} 2 \text{ Fe S} \\ 2 \text{ Zn S} \end{smallmatrix} \right] \text{Sn 2 S} + 2 (\text{Cu S}) + \text{Sn 2 S}$ —with Antimony, Silver, Iron, and Zinc, in *Weissgilitigerz*, a mechanical mixture of *Brittle Sulphide of Silver* with *Grey Antimony*, *et cetera* ; with the Sulphides of the same Metals and Sulphide of Arsenic, in *Grey Copper* ; with Antimony and Silver, in *Antimonial Grey Copper* ; and with Lead, Antimony, and a little Iron, in *Bourbonite* ; with Silver, a little Iron and Arsenic, in *Gansekothigerz* ; with Bismuth, in *Cupreous Bismuth* ; and with Bismuth, Lead, a little Nickel, and Tellurium, in *Siberian Needle Ore* ;

d. In an Oxidized state, either alone or in combination with other Metallic Oxides and Water, namely : as Suboxide, in *Red Oxide of Copper*—*Rothkupfererz*— $[\text{Cu}^2 \text{ O}]$; as Oxide with Sesquioxide of Manganese and Water, in *Cupreous Manganese*—*Kupfer-manganerz*— $\left[\begin{smallmatrix} \text{Cu O} \\ 3 (\text{Mn}^2 \text{ O}^3) \\ 3 (\text{H O}) \end{smallmatrix} \right] + 3 (2 \text{ Mn}^2 \text{ O}^3 + 3 (\text{H O}))$; and as an Oxide, with a little Peroxide of Manganese, Iron, and Water, in *Native Oxide of Copper* $[\text{Cu O}]$;

e. As an Oxide, in combination with Chloride of Copper in *Atacamite* $[\text{Cu Cl} + 3 (\text{Cu O}) + 6 \text{ aq}]$;

f. In an Oxidized state, with Acids, either alone or with other Metallic Salts, or Earths, and Water, thus :—

¹ With Carbonic acid and Water, in *Blue Carbonate of Copper*—*Azurite*— $[2 (\text{Cu O}, \text{C O}^2) + \text{Cu O}, \text{H O}]$; and in *Malachite* $[2 (\text{Cu O}) \text{C O}^2 + \text{H O}]$;

² With Arsenic Acid and Water, in *Condurrite* $[6 (\text{Cu O}) \text{As O}^5 + 4 \text{ aq}]$; in *Euchroite* $[4 (\text{Cu O}) \text{As O}^5 + 7 \text{ aq}]$; in *Erinite* $[5 (\text{Cu O}) \text{As O}^5 + 2 \text{ aq}]$; in *Prismatic-Copper Mica* $[8 (\text{Cu O}) \text{As O}^5 + 12 \text{ aq}]$; and also with Alumina, in *Liroconite*—*Octohedral Arseniate*— $[2 (\text{Al}^2 \text{ O}^3, 3 \text{ H O}) + 3 (4 \text{ Cu O} \text{As O}^5, 8 \text{ H O})]$;

³ With Phosphoric Acid and Water, in *Libethenite* $[4 \text{ Cu O}, \text{PO}^5 + 2 \text{ aq}]$; and in *Phosphoro-chalcite*—*Pseudo-Malachite*— $[5 \text{ Cu O}, \text{P O}^5 + 5 \text{ aq}]$;

⁴ With Sulphuric Acid and Water, in *Native Blue Vitriol* $[\text{Cu O}, \text{S O}^3 + \text{H O} + 4 \text{ aq}]$;

⁵ With Chromic Acid and Chromate of Lead, in *Vauquelinite* ;

⁶ With Silicic Acid and Water, in *Diopase* ($3 \text{ Cu O}, 2 \text{ Si O}^2 + 3 \text{ aq}$) ; with Silicic Acid, Water, and a little Carbonic Acid, in *Chrysocolla* ; and with Silicic Acid, and Silicate of Alumina, in *Allophane*.

Further, Copper is not only found *per se*, in scorise, *et cetera*, produced in the smelting of Cupreous Ores, but very often as a secondary constituent in the Slags from furnaced Argentiferous and Plombiferous minerals. It is found :—

^a Metallic, in pure *Gaarkupfer*,—the product of the third smelting of Cupriferous Ores—and in combination with other metals, in *Schwartzkupfer*—second product ; in the *Frischstücken*—the Argentiferous Leads, obtained in the refining of Copper Ores for Silver ; in the *Saigerdörnern*—the residues which remain after extracting the lead from the *Darrlinge*, and which yield a crude copper on being subjected to a process of smelting ; in the *Darrlingen*—eliquated coppers, from which the silver has been sweated out by lead ; and in *Cupriferous Workable Lead* ;

^b With Sulphur, *per se*, and also with Sulphides, *exempli gratia*, in *Rohstein*, *Bleistein*, *Kupferstein*, *Kupferleg*—one of the products produced by smelting the roasted *stein*, which is obtained by smelting roasted *Bleistein* with Quartzose Copper Ores, with Bleistein Slags, and Quartz—and in different *Tuttys* ;

^c In an Oxidized and Vitreous state, in all the slags which are obtained in the manufacture of Crude Copper, and in the refinement of Copper for Silver—Copper is also met with in *Fahlerz*, *Kupferkies*, *Kupferwismuthierz*, *Zinnkies*, *Cuban*, *Nadelerz*, *Cuproplumbite*, *Polybasite*, *Brochantite*, *Dihydrate*, *Ehlite*, *Tagalite*, *Thrombolite*, *Chalkolite*, *Mysorine*, *Aurichalcite*, *Klinoklas*, *Olivinite*, *Volbortite*, and *Kieselkupfer*.

Examination for Copper.

This examination is very simple, and so certain, that its presence or absence, in any combination, can be determined in a short time.

The combinations of copper with other metals, as met with in nature, generally contain selenium ; if exposed for some time to the oxidating flame on charcoal, and the metallic globule remaining treated with borax in a continued oxidating flame, the glass generally exhibits the color produced by oxide of copper. On melting the cooled glass after separation from the excess of metal, on another part of the charcoal in the reducing flame, it assumes on cooling, a red color, and is quite opaque. The latter does not, however, always succeed, because if exposed too long to the reducing flame, the copper separates, and the glass appears colorless. It succeeds better if a small piece of metallic tin is added, and only treated for a few moments in the reducing flame. A part of the tin oxidizes at the expense of the oxide of copper, and dissolves colorless in the glass, while the copper is reduced to a suboxide, which colors the glass red on cooling, and is opaque. The red color appears fainter in proportion to the other metallic oxides dissolved with it.

Copper is detected in this manner, in most furnace products, consisting only of metallic combinations. Such a combination is treated with borax or microcosmic salt, on charcoal in the reducing flame ; the glass pearl, while yet liquid, is removed from the metallic globule by the aid of the forceps, the color, if any, from oxide of copper observed, and the assay then placed upon another part of the charcoal, and treated as above with tin. If only a trace of copper be present, which can only occur with *Workable Lead*, a red-colored pearl is not always obtained, and if the metallic compound at the same time contains antimony, the glass on cooling becomes opaque, and is colored grey or black. In such a case, the metallic mixture must be previously melted *per se* on charcoal, in the oxidating flame, until all the antimony is volatilized, and the greater part of the lead is then dissolved in boracic acid, as will be given with the quantitative examination for Copper, under the process for refining ; the globule remaining is treated first with microcosmic salt in the oxidating flame, and the glass bead is then fused with tin in the reducing flame. If a trace of copper is present, the glass will be colored distinctly red on cooling, and will be opaque, entirely, or in separate portions.

Should the metallic combination contain much nickel, cobalt, iron, and arsenic, the greater part of the cobalt and iron can be removed by treatment with borax in the reducing flame, on charcoal, and recognized by the color of the glass, as given with iron. Lead is then added to that which has been obtained in a molten state in the reducing flame, by which means the greater part of the arsenic volatilizes, and by treatment with boracic acid, the lead, with any residual cobalt and iron, is dissolved. The remaining cupriferous nickel globule, and probably a part of the arsenic, is treated with microcosmic salt in the oxidating flame, and the observation made as regards the color which the glass assumes. If copper be present, it appears, while hot, dark green ; on cooling, clearer ; and when perfectly cold, bright green. The latter consists of the light brown of the oxide of nickel, and the blue of the oxide of copper.

The combinations of copper with sulphur, and other metallic sulphides, are either heated alternately, in the oxidating and reducing flames, on charcoal, till the sulphur is perfectly dissipated ; or treated with soda on charcoal, in the reducing flame, by which means the copper is obtained in a metallic state ; or dissolved in borax or microcosmic salt, and the glass treated with tin on charcoal, when the presence of copper is recognized by its red tinge. If the roasted substance contains, besides copper, other easily reducible metallic oxides, no pure copper will be obtained by the reduction with soda, but an admixture with other metals ; which, if not obtained in a single globule, must be sifted with water in a mortar, to remove the recrementitious particles, and lead added, if not already present ; it is then refined, by treating with boracic acid on charcoal. If convenient to dispense with the refining, the reduced metallic compound may be examined for copper with borax or microcosmic salt, as already mentioned with the metallic combinations. If the roasted assay contains, besides copper, oxide of iron only, a mixture of both metals is not obtained by the reducing process, but particular reguli of copper and iron, which, after purification, are readily distinguished by the lens or magnet. But if it contain oxide of tin, which occurs with tin pyrites, a white friable metallic mixture

is obtained by the reduction ; which, if melted for some time with a microcosmic salt pearl, in the oxidating flame, produces an opaque red glass. If not perfectly roasted, a mixture of sulphide of copper, and other metallic sulphides, are very readily obtained.

On treating the roasted substance with borax or microcosmic salt—even if it contains other metallic oxides, with the exception of oxide of bismuth and oxide of antimony—in the oxidating flame, and then in the reducing flame, after the addition of tin, the reaction of copper is always detected, if an appreciable quantity be present. But if it contain at the same time much bismuth or antimony, the glass assumes a dark grey color on cooling, which destroys the red tinge produced by suboxide of copper. If the amount of the bismuth or antimony present be small, the glass will often be colored brownish-grey. If a black or grey pearl is obtained, the roasted substance must be mixed with soda, borax, and proof lead, and this compound melted in the reducing flame. The metallic globule obtained by this means must first be treated *per se* on charcoal, and then with boracic acid, until either a pure globule of copper is obtained, or till the whole is dissolved, when the copper communicates a blue, green, or red color to the boracic acid ; or the globule of copper, freed by boracic acid from the greater part of lead and bismuth, is treated with microcosmic salt and tin, as already mentioned. The latter procedure, which volatilizes the antimony, and separates the bismuth perfectly by boracic acid, is the most certain.

If the copper in a substance is so insignificant that a portion, dissolved in borax or microcosmic salt, produces with tin no cupreous reaction, a greater quantity, about 100 milligrammes, must be roasted, as with a quantitative copper examination ; the roasted substance is then to be mixed with equal parts of soda and borax, and if it contains no easily reducible metal, 30 to 50 milligrammes proof lead added, and reduced as in a quantitative examination of the copper. The metallic mixture obtained as a globule by this means, in which all the copper is contained can be examined further for copper, after treating with boracic acid, and then with microcosmic salt and tin. The subsequent method of pro-

cedure has already been mentioned. The protoxide and suboxide of copper, as met with in nature, and also the latter in combination with other metallic oxides, behave *per se* towards borax and microcosmic salt, and with tin in the reducing flame, perfectly similar to the oxide of copper; and thus the presence of copper can be readily recognized in the minerals. The copper can be separated also from these minerals by a simple reducing process, with soda and a small portion of borax. The native copper salts, which include silicate of copper and a compound of this, with silicate of alumina—Allophane,—impart to borax and microcosmic salt the color of protoxide of copper, particularly if treated with tin, on charcoal in the reducing flame. If it be necessary to separate the copper in a metallic state, the sulphate of copper must be previously roasted slightly, in the oxidating and reducing flames alternately; this is reduced, like the other salts, with soda and borax on charcoal; the copper now generally coheres, forming a globule, while the difficultly reducible metallic oxides are dissolved by the borax.

In manipulation with sulphate or arseniate of copper, nickel, cobalt, or iron, the sulphur volatilizes by roasting, but part of the arsenic remains with the oxide of nickel, as arseniate. If the roasted substance be reduced with soda and borax on charcoal, the copper, nickel, and arsenic produce a fluid metallic globule, and oxides of cobalt and iron are dissolved by the borax. If the reduced metallic bead contains copper, it must impart a green tinge of both nickel and copper to borax or microcosmic salt in the oxidating flame, which becomes somewhat paler on cooling.

The presence of copper can also be determined, if treated with tin, by such a glass losing its transparency on cooling, and assuming a red tinge. If the borax glass, without the addition of tin, be treated in the reducing flame till all the nickel and copper is separated in a metallic state, and if then the glass be observed to appear blue, the roasting will have occurred imperfectly, and by the reduction some arsenide of cobalt has accompanied the metallic button.

It is difficult, by borax or microcosmic salt, to determine the

copper in slags as protoxide or suboxide, with the exception of that contained in *Gaarkupfer*, on account of the small quantity generally present; and, moreover, the other ingredients, which are chiefly silicates of different earths and difficultly reducible metallic oxides, destroy the reaction of oxide of copper. For this reason, instead of employing the reduction process, the slags must be treated with soda on charcoal. If even by this method, also, copper should not be detected, a greater quantity, about 100 milligrammes, must be reduced with equal parts of soda, the half of borax, and 30 to 50 milligrammes proof lead, and the lead, united to a globule, treated with boracic acid till all is dissolved, or the copper is concentrated. If the slag contains a trace of copper, this becomes reduced, and combines with the lead, and in the first case, has colored the boracic acid red, green, or blue. If the copper present is very minute, the tinge is seen on those parts only where the latter part of the lead containing copper was dissolved. If the slag contains one per cent. of copper, and the glass be treated in the reducing flame, the lead only is dissolved, and the copper remains in a melted state, with its peculiar greenish blue color. If the copper be exposed some time to the oxidating flame, it becomes oxidized, and the whole glass is colored red, by the suboxide formed. In the second case, the metallic globule, melted with boracic acid, is treated with micro-cosmic salt and tin, as above.

A small quantity of copper contained in a substance can often be detected, if not in combination with sulphuric acid, by one or two drops of hydrochloric acid. It is only necessary to moisten the substance with this acid, and heat it in the forceps, in the apex of the blue flame, when, by this means, the outer flame is colored greenish-blue, and often reddish-blue, by the chloride of copper formed. The coloring is more beautiful and stronger, the richer the substance is in oxide of copper. Silicates, *exempli gratia*, slags, must be pulverized as finely as possible in a mortar, this powder moistened with a drop of hydrochloric acid in a porcelain basin, dried over the flame of a lamp, and the dried powder kneaded into a granular mass with a drop of water. This grit is placed in the ring of a platinum wire, and melted in the apex of

the blue flame. If the silicate contains copper, a blue coloring ensues in the outer flame. If the mineral containing copper be heated, *per se*, in the apex of the blue flame, with the exception of Atacamite, the outer flame is colored beautifully green. If the mineral, at the same time, contains much lead, a blue flame, with greenish streaks, results.

§ 12. *SILVER—Ag—Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Silver occurs in nature :—

a. Metallic, both *per se*, and in combination with other metals, namely, as *Native Silver* [Ag], which is often contaminated with minute portions of Antimony, Arsenic, and Iron ; in *Native Gold*, which contains more or less silver ; in *Native Amalgam* [Ag, 2 Hg] and [Ag, 3 Hg] ; with Antimony, in *Antimonial Silver* [2 Ag, Sb] and [3 Ag, Sb] ; with Gold and Tellurium, in *Graphio Tellurium* [Ag, Te + 3 (Au, 3 Te)] ; with Iron, Arsenic, and a little Antimony, in *Arsenical Silver*—which is probably a mixture, and not a peculiar species ;—with Gold, Tellurium, and Lead, in *Yellow Tellurium—Weiss Silvanerz* ;—and with Selenium and Copper, in *Eukairite—Selenide of Silver and Copper* ;—

b. With Sulphur, both *per se*, and in combination with other sulphides ; thus, *per se*, in *Sulphide of Silver—Silver Glance*—[Ag S] ; with Antimony and a little Copper, in *Brittle Sulphide of Silver—Brittle Silver Glance*—[6 Ag S + 2 Sb 3 S] ; with Arsenic, Copper, and Antimony, in *Eugen-glance or Polybasite* $\left\{ \begin{smallmatrix} 2 \text{ Sb, } 3 \text{ S} \\ 2 \text{ As, } 3 \text{ S} \end{smallmatrix} \right\} + 9 \left\{ \begin{smallmatrix} \text{Ag S} \\ \text{Cu S} \end{smallmatrix} \right\}$; with Arsenic, and a very small proportion of Antimony, in *Sulphide of Silver and Arsenic*, the light red variety of *Red or Ruby Silver*—it is the *Lichtes Rothgiltigerz* or *Arsensilberblende* of the Germans [3 (Ag S), 2 As 3 S] ; with Antimony, in *Sulphide of Silver and Antimony*, the dark red variety of *Ruby Silver—Antimonsilberblende* of the Germans [3 (Ag S) 2 Sb, 3 S] ; with Antimony, a little Copper, and Iron, in *Myargyrite—Hemi-Prismatic Ruby Blende*—[Ag S, 2 Sb 3 S] ; with Copper and a little Iron, in *Sulphide of Silver and*

Copper—*Argentiferous Copper Glance*;—with Antimony, Copper, a little Iron, and Zinc, in *Weissgiltigerz* and *Graugiltigerz*; with Bismuth, Lead, together with a little Iron and Copper, in *Bismuthic Silver*; with Antimony, Arsenic, Copper, Iron, and Zinc, in *Grey Copper*; and as a minute constituent in most Lead and Copper ores, to which belong *Selenide of Lead*, *Copper Glance*, *Copper Pyrites*, *et cetera*.

c. In combination with Chlorine, in *Chloride of Silver*—*Hornsilver*—[Ag Cl]; and,

d. In combination with Iodine, in *Iodic Silver* [Ag 2 I].

Silver occurs in the products of smelting furnaces:—

¹ Metallic, both *per se* and in combination with other metals; namely, *per se*, as *Brandsilber*—*blicksilber* refined by cupellation—and *Amalgamated Silver*—the amalgam of silver and mercury obtained in extracting silver from its ores by the process of amalgamation; it generally contains copper, antimony, *et cetera*;—with a little Lead, and sometimes minute portions of Copper, in *Blicksilber*—the crude silver obtained in the refinement of lead for silver, after nearly the whole of the lead has been converted into Litharge;—with Lead, and also with Copper and traces of Antimony, Arsenic, Iron, and Sulphur, in the *Workable Lead*; as a secondary Constituent in the *Black Copper* obtained from Copper and Lead ores; and, also, in extremely small quantities, in *Abstrich Lead*—*Abstrich Lead* is the *Workable Lead* from which the *Abstrich* has been raked off.

² Combined with Sulphur and other metals, in which, however, it forms only a very minute constituent; namely, in the *Bleistein* obtained from Sulphide of Lead, and Sulphide of Iron; in the *Kupferstein*, from Sulphide of Copper, Sulphide of Lead, and Sulphide of Iron; in the *Rohstein*, from Sulphide of Iron, and, sometimes, a little Sulphide of Lead and Sulphide of Copper; and in the compound *Flue-rakings* from the smelting of different sulphides which yield Silver; as also, in a fine mechanically divided state, in the slags obtained by the smelting of Silver ores, or of the argentiferous products of their reduction.

³ In an oxidized state, but only in exceedingly small quantities, as in *Litharge*, in the *Abstrich*, and in the *Cupel Grounds*

from which the *Workable Lead* has been separated. Silver is found also in Tellusilber, Aquerite, Schriftez, Weisstellur, Tellurblei, Selensilber, Sprodglaserz, Xanthokon, and Sternbergite.

Examination for Silver.

Some of the minerals, alloys, and furnace products, previously mentioned, are so constituted as to be readily recognizable by their exterior appearance, for silver. To these belong Native Silver, Native Gold of a very light color, *Brandsilber*, *Amalgamated Silver*, and *Blicksilber*. Other argentiferous minerals, when combined with volatile ingredients, give pure silver beads when exposed to a strong oxidating flame, and the charcoal becomes coated with a red sublimate. These are Antimonial Silver, Arsenical Silver, natural and artificial amalgams. When the latter are heated in a glass matrass, mercury volatilizes and deposits in orbicles, which may readily be made to cohere, by tapping the vessel. The residuum, which is nearly freed from the mercury by this process, gives, when smelted on charcoal, a beautiful white globule. If Graphic or Yellow Tellurium be heated on charcoal, an auriferous yellow globule remains, which is composed of gold and silver; by treating this alloy with aqua regia, the gold dissolves and the silver deposits as a chloride. Native gold, containing silver, may also be treated in this manner, when separation is requisite. Those minerals which contain, besides volatile metals, copper, yield a cupreous silver bead when assayed upon charcoal in the oxidating flame. To obtain the silver from these in a pure state, they must be mixed with lead and cupelled.—Eukairite belongs to this class. Silver is detected in *Workable Lead*, *Abstrich-Blei*, and in impure *Black Copper*, by cupelling the first two, *per se*, and treating the last with ten times its volume of proof lead on charcoal. If it be requisite to examine at once for silver in minerals and furnace products, which consist of metallic sulphides, or only contain such, the quantitative silver examination with proof lead and borax is the most satisfactory method for this object. The necessary information upon this treatment will be given under the

above-mentioned examination. Native Chloride of Silver—Hornsilver—fuses on charcoal in the oxidating flame, to a brown, grey, or black bead, which, if pure, gives metallic silver in the reducing flame. This decomposition is immediately effected with soda on charcoal. Furnace products, which contain only a very small quantity of silver,—for instance, *Litharge*, *Cupel Grounds*, and *Abstrich*,—must be assayed according to the process given under the quantitative examination of such substances.

§ 13. *MERCURY—Hg—Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Mercury occurs in nature :—

- a. Metallic, *per se*, in *Native Mercury* [Hg] ; and combined with Silver, in *Native Amalgam* ;
- b. In combination with Sulphur, as *Cinnabar*, or *Sulphide of Mercury*, and in *Lebererz*, or *Hepatic Cinnabar* [Hg S] ; the latter is, however, contaminated with Carbon, Silicic Acid, Oxide of Iron, and other substances ;
- c. In combination with Chlorine, as *Chloride of Mercury* or *Horn Quicksilver* [Hg Cl] ; and,
- d. In combination with Iodine, as *Iodic Mercury*.

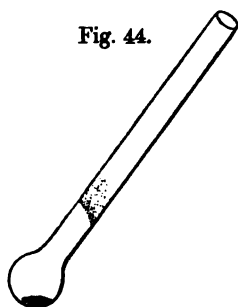
Mercury also forms a constituent of many products, residues, scorise, *et cetera*, of the *amalgamation process* for extracting Silver from its ores. To these also belong the products of the amalgamation of Gold and Silver Ores, namely, *Gold Amalgam*, *Silver Amalgam*, and the unwashed residue in which finely divided *Silver Amalgam* is generally present. If the Ores to be amalgamated contain Copper and Lead, these metals will be found in the residues, when the process is completed.

Examination for Mercury.

Native Mercury possesses all the properties of the pure metal, and need not therefore be more particularly examined, unless it be required to detect traces of other metals present.

The combinations of mercury with gold and silver, to which

belong both native and artificial amalgams, and also the impure residues obtained by the washing of silver, copper, and lead amalgams, should be ignited in a flask, or bulb tube—see Fig. 44—over the spirit-lamp. The mercury is separated, volatilized, and deposited in the form of small metallic globules in the colder part of the flask or tube, which cannot be confounded with any other metal. In the examination of the compounds of mercury with sulphur, chlorine, or iodine, to which Native Cinnabar, He-



patic Cinnabar, Horn Quicksilver, and Iodic Mercury belong, a portion of the substance should be triturated in an agate mortar, with an equal bulk of soda, the mixture introduced into the small glass vessel, and heated to redness over the spirit-lamp. The sulphur, or chlorine, combines at this temperature with the radical of the soda, forming sulphide or chloride of sodium, while metallic mercury becomes free, ascends as vapor, and condenses on the upper part of the flask in a greyish sublimate, which coheres into small metallic globules on gently tapping the tube. If the quantity of this metal present be so minute that no metallic sublimate of mercury is obtained, the experiment should be repeated in the same manner, introducing at the same time the end of an iron wire, covered with a piece of gold leaf, into the flask, and holding it a little over the surface of the mixture, when the gold becomes perfectly white, or in a great measure so, even when the quantity of mercury present is extremely small.

According to WACKENRODER, the haloid salts of mercury give a sublimate on charcoal. The oxygen salts also give, with chloride of sodium,—sulphide of mercury with a mixture of soda and chloride of sodium also,—a heavy or light white vapor and sublimate of subchloride of mercury. If a substance is to be examined for mercury, in which the latter is not combined with sulphur, and which, *per se*, gives no sublimate, it should be reduced to a fine powder, and fused with dry chloride of sodium on charcoal,

in the reducing flame ; a white sublimate of subchloride of mercury is procured. If the mercury, however, be combined with sulphur, the substance must be fused with a mixture of soda and chloride of sodium.

As chloride of sodium also yields a white sublimate, *per se*, on charcoal, in the reducing flame, but which appears later than the sublimate of subchloride of mercury, and then only when the heat is very strong, the behavior of chloride of sodium, *per se*, on charcoal, should be first studied, before an examination for mercury in this way be undertaken.

§ 14. PLATINUM—Pt—PALLADIUM—Pd—RHODIUM—
—R—IRIDIUM—Ir—OSMIUM—Os—*Presence in the*
Mineral Kingdom.

These Metals generally occur united together ; sometimes, also, with traces of Iron, Copper, and Lead, in *Native Platinum*, in which Platinum is, however, the principal constituent.

Palladium is likewise found native, combined with a little Platinum and Iridium, along with the *Native Platinum*, in Brazil.

Rhodium occurs only as an extremely small constituent in *Native Platinum*.

Iridium occurs native; combined with a little Osmium, along with *Native Gold*, and *Native Platinum*, in Russia. It is also found, with a larger proportion of Osmium, in peculiar grains—*Osmium-Iridium*—[Ir Os], along with *Native Platinum*, in South America. The quantity of Osmium in *Osmium-Iridium* varies greatly.

Osmium has been found, as yet, only in the last named compound, and in *Native Platinum*.

Behavior of the preceding Native Metals before the Blowpipe.

These metals cannot be so separated from one another by the Blowpipe, that each metal may be recognized when treated with borax or microcosmic salt, on a platinum wire or on charcoal,

since they neither oxidize nor dissolve ; beads, more or less colored, are in this case obtained ; but the color proceeds from the mixed oxidizable metals, namely, from copper, iron, *et cetera*, which may be readily detected in this way, in such combinations.

If they be fused with lead, and the alloy subjected to refinement in a cupel,—which operation lasts only as long as it affords the difficultly fusible metals—an infusible metallic compound is at length obtained, which contains, proportionably, much lead ; but if a sufficiently large grain of gold be added to it, and then refined in a strong heat, a yellowish-white, or even a platinum-grey metallic button, perfectly free from lead, will be obtained, in case too much platinum or iridium is not present in the alloy. If it is not obtained of a *fine* quality from the cupel, it may be, very readily, with boracic acid, on charcoal, in the oxidating flame.

The alloy of gold, platinum, iridium, rhodium, palladium, *et cetera*, thus obtained, can only be analyzed in a moist way when it is required to recover the gold. For this purpose it should be dissolved in aqua regia, the solution partly evaporated, diluted with weak alcohol, and the platinum and iridium thrown down from this solution by chloride of potassium or chloride of ammonium, and the gold, by a fresh prepared solution of protosulphate of iron. The latter is obtained in the metallic state, and requires only to be filtered,edulcorated, and fused with a little borax, on charcoal.

Osmium-Iridium is the only compound which can easily be decomposed, and in which osmium may be recognized. When it is strongly ignited in a glass flask, with saltpetre, oxide of osmium is formed, and may be recognized by its foetid smell, which is similar to that of chloride of sulphur.

§ 15 GOLD—Au—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Gold always occurs, in nature, in the metallic state, thus :

a. Mixed with other metals, as with, more or less, Silver, in *Native Gold* ; with Tellurium and Silver, in *Graphic Tellurium* ;

with Tellurium, Lead, and Silver, in *Yellow Tellurium*; with a large quantity of Tellurium and a little Iron, in *Native Tellurium*; with Lead, Tellurium, a little Copper, Silver, and Sulphide of Lead, in *Black Tellurium*; in a combination of Selenide of Molybdenum with a little Silver, in *Noble Molybden Glance*.

b. In metallic Sulphides; namely, in many Iron and Copper Pyrites, but, however, in small quantities. The quantity of Gold in these Pyrites is very different. The Iron Pyrites so widely diffused in Saxony, which lie in contact with the gangues, and which, at the same time, contain Sulphide of Antimony, and finely disseminated Ruby silver, or other Silver Ore, always yield more Gold than those occurring in the same situation in which no Antimony can be detected: the former contains, in a hundredweight, owing to the admixture of Silver Ores, from five to ten ounces of silver, eight ounces of which yield from 0.5 to 0.8 of a grain of Gold; and the latter contain, in the hundredweight, from 0.125 to 0.5 of an ounce of Silver, eight ounces of which yield only from 0.15 to 0.18 of a grain of Gold.—It is better always to control the assay of silver by analysis in the humid way. For assay, fuse 200 grains of the finely pulverized ore with litharge, carbonate of soda, and charcoal, and cupel the produced button on bone ashes. The results are generally a trifle too low. For humid analysis, fuse 100 grains of the ore with about 300 of carbonate of soda; treat the fused mass with water, to dissolve out the soluble chlorides, precipitate by hydrochloric acid, wash the chloride of silver, weigh, and calculate the amount of silver.

Gold Amalgam is the only product of smelting furnaces, extensively occurring, in which Gold forms the principal ingredient.

However, as auriferous Silver Ores are often smelted, Gold is also found as a secondary constituent, in many products obtained in the reduction of silver; more particularly in the following:—*Brandsilber*—if the Gold has not been already separated, *Blicksilber*, *Amalgamated Silver*, *Workable Lead*, *Black Copper*, *Bleistein*, *Kupferstein*, and *Rohstein*.

Examination for Gold.

Native Gold is easily recognized by its peculiar color. The remaining auriferous metals, Pyrites only excepted, should be treated, when the volatile metals are also to be detected, on charcoal in the reducing flame, until an unchangeable metallic button is obtained. With some minerals, *exempli gratia*, Graphitic Tellurium, Yellow Tellurium, and Black Tellurium, a button remains, which, on cooling, possesses the color of gold, and contains the silver present in the mineral. If it should happen that a pure metallic button is not obtained, a little proof lead and borax must be added to the remaining mass, and the whole treated for some time in the reducing flame. The easily reducible metals combine with the proof lead, and, after cooling, can be separated from the gold and silver by cupellation on bone ashes. If the button obtained by cupellation does not possess the color of gold, but appears white, the quantity of gold is less than that of silver, in which case, the button should be placed in a porcelain capsule, a few drops of nitric acid poured on it, and the capsule heated over the lamp.

If the button does not contain more than a fourth part of its weight of gold, it becomes completely black, and then decomposes, the silver being dissolved, while the gold remains in black flakes. When the silver button contains more than the fourth of its weight of gold, it is blackened, but the silver is not dissolved. It is neither blackened nor dissolved, if the proportion of gold to silver is nearly equal : in this case the button must be melted with twice its bulk of pure silver, on charcoal, and again treated with nitric acid, by which the mass becomes black, and dissolves ; the pure gold being left behind.

Gold Amalgam is treated, first in a flask similar to the Silver Amalgam, in order to drive off the greater part of the mercury, and then on charcoal in the oxidating flame, when a button of pure gold is obtained.

Brandsilber, as also *Blicksilber* and *Amalgamated Silver*, are treated, after being refined with a slight addition of proof lead, on a cupel with nitric acid, and examined whether the silver becomes

black, and whether black gold flakes, or black particles, remain after the solution of the silver.

Workable Lead is refined *per se*, and *Black Copper*, with an addition of proof lead on the cupel, and the resulting metallic button treated with nitric acid. If the silver becomes black, or if black particles remain after the silver is dissolved, the metals contain gold.

Iron and Copper Pyrites, *Bleistein*, *Kupferstein*, and *Rohstein*, must be first examined for silver, in the same manner as in the quantitative examination. The resulting button of silver is to be placed in a porcelain capsule, containing some hot nitric acid, and *quickly* examined with a lens, whether it dissolves with a black or white color. In the former case, the substance contains gold ; in the latter, it is absent. When a button is not obtained in the examination of Pyrites, it does not follow that gold is not present, as the quantity is often so minute that it cannot be recognized on the cupel with the lens, even from 100 milligrammes of ore. Hence, two or more fragments should be subjected to examination, the resulting *Workable Lead* concentrated by cupellation, mixed with a little pure silver, and again refined, by which means the gold is combined with the silver, and may be recognized as above, by the black color of the button, when treated with hot nitric acid.

§ 16. TIN—Sn—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Tin occurs in nature in the following minerals :

In combination with Sulphur, and Sulphides of Copper and Iron, in *Tin Pyrites* ; as an Oxide, in *Tin Stone—Pyramidal Tin Ore*—[Sn O²], which contains traces of Iron, Manganese, Tantallic Acid, and Silica ; as a constituent in most Tantalites, and numerous other minerals, containing Titanium and Uranium.

It very rarely occurs in furnace products, unless Tin Ores have been smelted, and then portions of it will be found in the slags. When Tin Pyrites accompany an ore of Copper, and cannot be separated in a pure state by reduction, a quantity of Tin will necessarily be detected in the first products of the copper smelting, particularly in the *Rohstein*.

Examination for Tin.

Tin is readily recognized in Tin Pyrites, by exposing a small piece of the mineral to the oxidating flame, on charcoal. The assay at first exhales a sulphurous acid smell, afterwards becomes snow white on the exterior, and a white coating is perceivable on the support surrounding the specimen ; this sublimate is so profuse, that the charcoal is not seen in any part between it and the metallic bead. This deposit is not expelled in either flame ; in other respects, its comportment is similar to the oxide of tin.

The tin can be separated from this mineral, in the metallic state, by roasting alternately in the oxidating and reducing flame, then pulverizing the mass in an agate mortar, with double its quantity of a mixture consisting of 100 parts soda, 50 parts borax, and 30 parts silica, and heating the whole on charcoal in the reducing flame, until the tin and copper are reduced to a globule. In this treatment the whole of the copper becomes reduced, but only a portion of the tin, the rest remaining dissolved in the glass with sesquioxide of iron. The cupriforous tin bead, which is friable if too small a quantity of tin is not present, must be separated, and what remains treated with soda in a strong reducing flame, by which means the rest of the tin becomes reduced, and is obtained in particles, by pulverization, and sifting with water. The cupreous globule, when heated upon charcoal with microcosmic salt in the oxidating flame, fuses, and the resulting glass, when cold, has a reddish color, owing to the presence of suboxide of copper.

With the stanniferous *Rohstein*, the treatment is the same as above. Tin Stone behaves like the oxide of tin, with this difference, that it imparts the color of iron to borax and microcosmic salt, and often affords the manganese reaction with soda on the platinum wire.

The best method for the detection of tin in Tantalites and Tin Slags is by reduction with soda ; but in such a case it is necessary to add a small portion of borax, to dissolve the tantalic combinations, and prevent the reduction of the iron. After the completion of such a process, the tin is obtained by pulverization and

sifting. To be convinced that the metallic particles obtained are tin, dissolve protoxide of copper in microcosmic salt, add some of them to the flux, and then heat the whole upon charcoal in the reducing flame. If tin is present, the glass will be colored reddish on cooling.

§ 17. *ANTIMONY*—Sb—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Antimony is found in nature :—

a. Metallic, combined with other metals ; namely, with a little Silver and Iron, in *Native Antimony* [Sb] ; with Silver, in *Antimonial Silver* ; and with Arsenic, in *Arsenical Antimony* ;

b. With Sulphur, and Sulphides of other metals ; *exempli gratia, per se*, in *Grey Antimony*, which generally contains Lead, Copper, Arsenic, and Iron ; with Iron, in *Berthierite* ; with Nickel and Arsenic, in *Nickeliferous Grey Antimony* ; with Lead and traces of Iron, Copper, Bismuth, and Zinc, in *Jamesonite* ; with Lead, Copper, and Iron, in *Bournonite* ; with Lead, and a trace of Copper, in *Zinkenite* ; with Lead in *Antimonial Lead Glance* ; with Silver and Copper, in *Melan-Glance* ; with Silver, Copper, and Iron, in *Miargyrite* ; with Silver, in *Dark Ruby Silver*—*Rhombohedral Ruby Blende* ;—sometimes with Silver and Arsenic, in *Light Ruby Silver* ; contaminated with Arsenic, Silver, and Iron, in *Arsenical Silver* ; also, more or less, in the following argentiferous minerals : namely, with Silver, Copper, Iron, and Zinc, in *Weisagiltigerz* and *Graugiltigerz* ; with Copper and Silver, in *Antimonial Grey Copper* ; with Copper, Arsenic, Silver, Iron, and Zinc, in *Grey Copper*—*Tetrahedral Copper Glance* ;—and in a very minute quantity. with Copper, Iron, Silver, and Arsenic, in *Kupferblende* ;

c. As an Oxide, in *White Antimony* [Sb O³] ; which is sometimes contaminated with Sesquioxide of Iron ; and with Sulphide of Antimony, in the *Red Antimonial Ore* [Sb O³ + 2 (Sb, 3 S)] ;

d. As Antimonious Acid, in *Antimonial Ochre* [Sb O⁴]. Antimony forms a small ingredient in many Argentiferous and

Plumbiferous furnace products, when the smelted or amalgamated ores are not free from Antimonial Silver or Lead Ores. This class includes *Workable Lead*, *Amalgamated Metals*, and the *Abstrichblei*, which hold it in a metallic state ; further, the *Rohstein*, *Bleistein*, *Kupferstein*, and *Lead Rakings*, in which it is found as a Sulphide ; and the *Abstrich*, in which it exists as Antimonious Acid, in combination with oxide of lead. Antimony is also met with in Plagionite, Fahlerz, Geokronite, Kilbrickenite, Kobellite, Antimonbloom, Antimonocker, and Romeite.

Examination for Antimony.

The examination for antimony is not very difficult, as it can be detected in most combinations in which it occurs in a metallic state, by two methods ; namely,—

a. Upon charcoal ; when the antimony volatilizes, and the support is coated with a white sublimate ; and,—

b. In an open glass tube ; when antimonial fumes are evolved, consisting of oxide of antimony and antimonious acid, which condense in the upper part. If the treatment occurs with metallic compounds, or metallic sulphides, in which the antimony is to be sought for,—as, for example, in the minerals above mentioned, where the antimony is contained as a metal, and in furnace products, the *Workable Lead*, the *raw amalgamated metals*, the *Abstrichblei*, the *Roh-*, *Blei-*, and *Kupferstein*, and the *Tutty*,—the examination according to the first method must be performed as follows : A fragment of *Workable Lead*, *raw amalgamated metal*, or *Abstrichblei*, is taken,—the other products and minerals are best employed in a powdered state,—and placed in a smooth cavity made in the charcoal, and then submitted to a *weak* reducing flame. By holding the support in an horizontal position, if any arsenic be present, it will volatilize, if not in combination with nickel, and coat the charcoal with a white or grey sublimate, at a great distance from the assay. Should no arsenic be present, there will be produced a slight deposit of oxide of antimony. When no more arsenical vapor is evolved, the flame is to be directed upon the sublimate, without igniting the charcoal ; by

this procedure the whole of the arsenic is expelled, and a clear surface remains, upon which the antimonial deposit is recognizable. If an operator treats a powdered assay in the reducing flame, it may cohere into a bead ; but this will only occur when the substance is very fusible. When the latter is the case, the oxidating flame must be brought into action. This deposit is white, when no lead is present, and can be driven from place to place, either by the oxidating or the reducing flame ; if the reducing flame is employed, it assumes a slight dark blue appearance, which is characteristic of the presence of antimony. If the substance contains lead, a yellow sublimate of the oxide of this metal is also obtained, which rests at a greater distance from the assay than the antimonial one, and when in thin layers, appears bluish, thus resembling a sublimate of antimony in the same disseminated state. When much antimony is present, the lead does not impede the determination, but if, on the contrary, the sample contains a large amount, an operator must not blow for too long a time upon it ; if this precaution be observed, only a white antimonial sublimate results, as the lead is not expelled unless by a long uninterrupted blast. The deposition of lead may be entirely prevented by the addition of some vitrified boracic acid to the substance under examination ; when this mixture is acted on in the reducing flame, the oxide of lead formed is absorbed by the boracic acid, while the greater portion of the antimony volatilizes, and coats the charcoal with its oxide. The antimonial lead globule must not be kept in the centre of the molten mass, but only in contact with it, because the antimony, in the former case, is vaporized with difficulty. If the substance contain zinc, a sublimate of this metal is also obtained, which is readily distinguished from that of antimony, by not being volatilized in the oxidating flame.

Minerals and furnace products containing antimony, as an oxide or acid, can also be partly investigated in the above manner, but a distinct sublimate is not always procured ; particularly if the oxide of antimony, or antimonious acid, be disseminated or combined with other bodies. When this occurs, the assay should be mixed with soda, and treated on charcoal, in the reducing flame, when the antimony volatilizes and coats the support with

an oxide, which is very distinct, even when a minute quantity of antimony is contained in the sample. If the antimonious acid be in combination with much oxide of lead, which is the case with the *Abstrich*, the manipulator must not blow for any length of time, so as to prevent the volatilization of much lead. Combinations of oxides of tin and antimony, or antimonious acid, which do not occur in nature, must be treated with a mixture of soda and borax, on charcoal, in the reducing flame. The oxides are reduced and separated in small limpid metallic globules, which must be sifted from the recementitious particles, and then heated on charcoal, with three times their volume of proof lead, and a small portion of vitrified boracic acid. If the assay be treated in the reducing flame only, antimony volatilizes, and coats the charcoal with a sublimate; the tin oxidizes, and covers the metallic button with a deposit, which dissolves with a part of the lead in boracic acid. The antimonial sublimate is freed from oxide of lead by this procedure, and can therefore be readily recognized. The oxide of tin, which deposits on the molten button, resembles the formation of oxide of nickel upon a nickeliferous lead globule; but as tin and nickel behave perfectly different towards borax, it suffices when the presence of the former is determined.

The second method for the detection of antimony, when in a metallic state in its combinations, is the following:—The substance is heated in a glass tube, whereby the antimony oxidizes and sublimes, forming a white fume, the behavior of which varies, according to the different metals with which the antimony is in combination. If the metals are readily oxidizable, antimonious acid fumes will be expelled, which are very stable, and not acted upon by heat.

The fume which escapes from a silver or copper compound, partly passes off, and partly deposits on the upper part of the tube. That which escapes possesses an acid smell; but, if sulphur happens to be present, the odor of sulphurous acid will predominate.

If the substance contains lead, an exhalation also ensues, which is very dense. The portion which volatilizes may be regarded as pure oxide of antimony; and the nonvolatile portion, as antimonite of lead.

That this method is not applicable to the examination of readily fusible metallic alloys, is easily understood ; for, on melting, they flow out from the tube, which is held obliquely.

When the substance is in combination with sulphur, it escapes as sulphurous acid, which is recognized by its characteristic odor ; and in this case, if only a small portion of antimony be contained in the assay, antimonious acid will often be generated, and no oxide of antimony. If the substance contains much arsenic, the antimonial vapor will be very profuse ; but, as the crystals of oxide of antimony and arsenious acid combine, they cannot always be distinguished from their appearances. When this ensues, it is preferable to examine on charcoal, as previously described.

Combinations of oxides of antimony, or antimonious acid, and other bodies, cannot always be examined with certainty in an open glass tube : for antimony, it is always better to employ the procedure with soda, on charcoal.

§ 18. *MOLYBDENUM*—Mo—*Presence in the Mineral Kingdom.*

It is found—

a. Metallic, combined with Sulphur, in *Rhombohedral Molybdena Glance* [$\text{Mo } 2 \text{ S}$] ;

b. As Molybdic Acid [Mo O_3], *per se* as a yellow coating upon the Sulphide of Molybdenum, and with Oxide of Lead, in *Gelb-Bleierz—Molybdate of Lead*.—BOUSSINGAULT found, in *Basic Molybdate of Lead*, from Paramo-Rico—near Pamplona, in South America,—Lead, Molybdic Acid, Carbonic Acid, Hydrochloric Acid, Phosphoric Acid, Chromic Acid, Sesquioxide of Iron, Alumina, and Silica.

Examination for Molybdenum.

The molybdenum in Molybdena-Glance is detected by means of microcosmic salt upon the platinum wire. This flux appears at first not to dissolve any of the mineral, but, after a long-continued oxidating flame, the microcosmic bead assumes a greenish color, owing to molybdic acid. If the undissolved portion of the

assay be removed with a forceps from the molten bead, and what remains treated with a reducing flame, the green color will be very characteristic. Molybdic acid, before the Blowpipe, fuses on charcoal, volatilizes, and imparts to the flame a yellowish-green tinge ; and where the assay rested, a reddish shining metallic speck is observed. The acid deposits also a crystalline coating upon the charcoal. Sulphide of Molybdenum, and also the metal, give, without fusing, the same reaction. The green color imparted to borax by molybdic acid remains perfectly transparent, after exposure for a very long time to the oxidating flame ; therefore the operator may rest satisfied that this reaction proceeds from no other metallic oxide.

The Native Molybdic Acid from Bisberg comports itself in a similar manner to the pure acid, but when treated with soda upon charcoal, it is imbibed, and peroxide of tin remains behind.

Gelb-Bleierz is submitted to the same treatment as the Molybdena Glance, for the detection of the molybdic acid ; with this precaution, that too much of the specimen be not added, or otherwise a black opaque glass will result.

MERLET's moist method for the detection of molybdenum in the foregoing minerals, is the following : powder the specimen, and fuse with nitre in a platinum spoon ; molybdate of potassa is formed. Dissolve in a porcelain basin with water over a spirit-lamp ; decant off the clear solution into another vessel, in order to get rid of the residuary matters that perhaps may occur ; and then treat the hot aqueous solution with hydrochloric acid, and immerse in the menstruum a strip of metallic copper. If only a trace of molybdic acid be present, the liquid, where the copper rests, will acquire a beautiful blue color, which disappears as the solution cools, but is immediately restored by heat. Metallic molybdenum, which is obtained by fusing the oxide in a crucible lined with charcoal, is not fusible before the Blowpipe ; but when heated upon charcoal, in the oxidating flame, it becomes oxidized ; and gradually coating, at no great distance from the assay, the support with a sublimate, which in many places, but particularly nearest the assay, is in transparent silky shining crystalline plates also in a pulverulent form. The deposit of molybdic acid is,

while hot, yellow ; and when cold, white. The crystalline plates appear the most beautiful when the assay is kept far from the Blowpipe flame ; but the heat must be properly regulated for their formation. The sublimate can be driven along the charcoal by the oxidating flame ; but the place which it leaves appears, when perfectly cold, dark copper-red, and shining oxide of molybdenum remains, which is produced by the molybdic acid coming in contact with the glowing charcoal. Molybdenum remains intact in the reducing flame.

Sulphide of Molybdenum—Molybdena Glance—which is also infusible, affords, in a continued strong oxidating flame, the same sublimate as pure Molybdenum, while its sulphur escapes as sulphurous acid.

Molybdic Acid fuses very readily, volatilizes in the oxidating flame, coating the charcoal with crystalline acid, and copper-red metallic shining oxide. Molybdenum possesses, in an oxidized condition, the property of tinging the outer Blowpipe-flame ; thus, if molybdic acid, in a moistened state, be treated on platinum wire with the apex of the blue flame, it volatilizes, coloring the outer flame yellowish-green, analogous to baryta. The same coloration is produced by Molybdena Glance, as may be seen by heating a thin fragment of this mineral, held in the platinum forceps, in the apex of the blue flame. No fusion takes place, but the outer flame assumes a yellowish-green color.

§ 19. *TUNGSTEN—W—Presence in the Mineral Kingdom.*

This element exists in nature as an Acid only, either *per se* [$W O_3$], or in combination with Bases, namely :—with Lime, in *Schwoerstein—Tungstate of Lime*— $[Ca O, W O_3]$; with Oxide of Lead, in *Tungstate of Lead—Scheelbleispath* ;—with Iron, and Protoxide of Manganese, in *Wolfram* $[Mn O, W O_3 + 3 (Fe O, W O_3)]$ —its composition has been represented by BERZELIUS as follows : $[Fe^2 O_3, 2 W O_3 + Mn^2 O_3, 2 W O_3]$; further, as a very small ingredient in some varieties of *Tantalite*, and in *Black* and *Brown Ytiro-Tantalite*.

Examination for Tungsten.

Tungstic acid is readily detected in Schwerstein, Wolfram, and Tungstate of Lead, in the following manner : Mix a small portion of powdered mineral with five times its volume of soda, and heat the mass strongly in a platinum spoon. Dissolve the fused assay in boiling water, and decant off the supernatant clear liquor, which contains tungstate of soda. If this solution be treated with hydrochloric acid, a white powder deposits, which, when heated, assumes a beautiful lemon color. The behavior of the tungstic acid in Schwerstein, towards microcosmic salt, has been previously given at page 120.

When the Tantalites are examined in this manner for tungstic acid, tantalic acid,—if the tantalum is contained as an acid,—will accompany tungstic acid, which prevents the yellow reaction when heat is applied to the mixture. Should this occur, the white powder must be separated from the solution by filtration, edulcorated, and treated with microcosmic salt, as follows : dissolve in the microcosmic salt upon a platinum wire, so much sesquioxide of iron that the glass produced will appear colorless or slightly yellow in the oxidating flame, and, when treated in the reducing flame, perfectly colorless when cold. Now add some of the moist precipitate, and submit the whole, first to an oxidating and then to a reducing flame. If tungstic acid be present, the glass will be tinged, either yellow, red, or blood-red, according to the quantity of the substance added.—As mistakes may arise, on account of titanic acid affording, with microcosmic salt, a similar reaction to tungstic acid, the examination should be extended. According to ROSE, tungstic acid gives a yellow glass with borax, and titanic acid, a violet one, which becomes *turbidly streaked*.—This reaction is so exceedingly accurate, that if a *trace* only of tungstic acid be contaminated with the tantalic acid, the ferruginous microcosmic glass will acquire a yellowish hue. The operator must exercise great care in adding the sesquioxide of iron, to avoid a blue reaction being obtained in the reducing flame. As nearly all Tantalites contain more or less sesquioxide of iron, this method will very seldom have to be employed, as the reaction

is speedily produced by dissolving the pulverized mineral in microcosmic salt, and treating in the reducing flame; if tungstic acid be present, a transparent dark red bead results. The presence of protoxide of manganese, oxides of tin, yttria, and lime, does not affect the coloration.—Tungstic acid is most conveniently obtained by decomposing the native Tungstate of Lime, finely pulverized, by hydrochloric acid; chloride of calcium is formed, and tungstic acid precipitates. Dissolved in ammonia, and precipitated again by acid, tungstic acid always forms a compound with the acid employed. It may be obtained in a separate state, by heating the tungstate of ammonia to redness. It is an orange-yellow powder, which becomes dull green when strongly heated. It is quite insoluble in water, and in acids, but dissolves in alkaline solutions.

Titanic acid, oxide of nickel, and antimonious acid, when combined with small quantities of iron, give similar reactions with microcosmic salt, as ferruginous tungstic acid.

§ 20. *VANADIUM—V—Presence in the Mineral Kingdom.*

It has been found in an *Iron Ore* from Taberg; a *Lead Ore* from Zimapan, in Mexico, also from Warloch-head in Scotland—which is principally *Vanadate of Lead*;—in *Volborthite*, *Pea Iron Ore*, from Steinlade, near Goslar; in *Mansfield Copper Slate*, *Hydrophite*, and *Vanadate of Lime*. The presence of Vanadium in *Pitchblende* most probably arises from the presence of *Vanadate of Lime*, the recently discovered mineral.

Vanadium is a very rare element.

Examination for Vanadium.

The pyrognostic properties of Vanadate of Lead are very characteristic. On charcoal, before the Blowpipe, it strongly decrepitates, fuses to a globule, then scintillates, giving a regulus of lead, and the support becomes coated with a yellow sublimate.

With microcosmic salt, it gives in the oxidating flame, a reddish-yellow, and, after cooling, a yellowish-green glass; in the reducing flame, it affords a beautiful chrome-green bead.

Per se, it fuses when held in the forceps, and presents, on refrigeration, its yellow tinge.

Vanadium, *per se*, on the platinum wire, in the oxidating flame, is converted into vanadic acid, which, with borax, gives, while hot, a dark yellow, and on cooling, a pale yellow glass. In the reducing flame, the glass is greenish or brownish, while hot, but, on cooling, chrome-green,—it is not colored blue by the addition of tin.

Vanadic acid, with microcosmic salt, dissolves readily, producing a reddish-yellow glass, which is, on cooling, pale yellow. It acquires a fine transparent green hue in the reducing flame, but the dark yellow color is recalled in the oxidation.

With soda it easily dissolves, and is absorbed by the charcoal.

On platinum foil, it fuses to a deep yellowish-red fluid, which becomes crystalline on refrigeration.

Vanadic acid is distinguished from oxide of chromium by producing a yellow glass on platinum wire in the oxidating flame, which is never the case with the latter.

The following is SEFTSTRÖM's method for obtaining the vanadic acid from slags or the minerals:—Fuse with an equal part of saltpetre, and two parts carbonate of soda,—when the temperature is very high, vanadate of soda is formed,—affuse the resulting mass with boiling water, and neutralize the liquid with nitric acid, then add chloride of barium, or acetate of lead, by which a precipitate of vanadate of baryta, or lead, is obtained; treat with sulphuric acid, and then separate the red liquid from the sulphate, digest it with alcohol, whereby, under formation of ether, the vanadic acid is resolved into the binoxide of vanadium, and the solution is blue; evaporate to dryness, and then heat the residue to strong redness to expel the sulphuric acid—impure vanadic acid remains; smelt it with nitrate of potassa until a portion of the cold sample loses its reddish appearance. Dissolve, filter, and immerse in the liquid a large piece of chloride of ammonium; a white precipitate of vanadate of ammonia will appear, which is insoluble in a saturated solution of the precipitant; filter and edulcorate, first with a solution of chloride of ammonium, and then with spirits of wine, of specific gravity 0.860; dissolve in boiling water, containing a few

drops of free ammonia, and, upon cooling, pure vanadate of ammonia is obtained, the salt from which all the other compounds of vanadium are formed.

From the solution of the vanadate of lead in nitric acid, the lead and arsenic can be separated by sulphide of hydrogen; the resulting blue solution of binoxide of vanadium, on evaporation to dryness, yields vanadic acid, with which pure vanadate of ammonia may be formed, by following the foregoing method.

§ 21. *TANTALUM*—Ta—*NIOBIUM*—Nb—*PELOPIUM*—Pp—
Presence in the Mineral Kingdom.

These three metals are only found as acids in combination with bases in the following minerals:—

Tantalite, *Columbite*, *Pyrochlore*, *Yttrio-Tantalite*, *Fergusonite*, *Samaraskite*, *Aeschynite*, *Euxenite*, *Polykras*, *Wöhlerite*, *Eukolite*, and in many *Tin-Stones*.

Examination for Tantalum, Niobium, and Pelopium.

Tantalic, Niobic, and Pelopic acids, when they constitute the chief ingredients of minerals, and when sufficient quantities of such are dissolved in borax, may frequently be recognised by giving an opaque or enamel-like glass by exposure to an irregular flame, particularly after treatment in the reducing.

Since ROSE has discovered niobium and pelopium in the *Columbite* of Bodenmais, the acids of which have several properties in common with tantallic acid, it is necessary, in the examination of those minerals which contain these acids, to seek a method for their complete separation. The surest way to effect this object with their very complex combinations, is to fuse a considerable quantity of the mineral, as finely pulverized as possible, with bisulphate of potassa, and the fused mass, after having been powdered, is dissolved in water. If the mineral contains tantallic acid, or niobic and pelopic acids, and also, perhaps, tungstic acid, these are separated by treatment with water, while any titanic acid present, as well as the bases, is dissolved.

The residue can either be fused with carbonate of potassa, or, when it is free from titanic acid and zirconia, treated at once with sulphide of ammonium, to separate the tungstic acid and oxide of tin. After filtration and a thoroughedulcoration, what remains is immediately treated on the filter with dilute hydrochloric acid to get rid of traces of iron, and now the operator ascertains whether he has to deal with tantalic, or niobic and pelopic acids.

For this purpose there are two ways : the first is, to examine the residuum, well washed, with microcosmic salt on platinum wire, as well as with cobalt solution on charcoal, to ascertain whether the microcosmic salt is tinged in the reducing flame, and what color the acid in question assumes by this treatment. The acid which, for example, separates from the Columbite from Bodenmais, or from the Pyrochlore from Miask, and which consists of niobic and pelopic acids, affords in the reducing flame a brown bead passing into violet, by which it is easily distinguished from tantalic acid, which occasions almost no coloration.

The second way depends upon some investigations made by ROSE. As, according to this chemist, tantalic acid is precipitated of a light yellow, pelopic acid of an orange-yellow, and niobic acid of a dark orange-red color from the solution of tantalate, niobate, and pelopiate of potassa in water, when it is acidulated with hydrochloric acid and then mixed with tincture of galls, the examination may be conducted in the following manner:—The residual acid in question is mixed carefully with five times its volume of carbonate of potassa in an agate mortar, and the mixture is converted, with a few drops of water, into a pasty mass, which is fused in small portions in the loop of a strong platinum wire to a clear bead, and every time the loop is full, the still hot bead is cast into a small porcelain dish. The fused beads are pulverized, the powder first treated in a porcelain dish with a little cold water, to dissolve the carbonate of potassa added in excess, and also niobate of potassa, if present, which appears to be more easily soluble in cold water than tantalate and pelopiate of potassa, and after the whole has been well stirred with a glass rod, the insoluble salts are allowed to deposit. If the supernatant

liquor appears clear, it is removed by means of a small pipette into another porcelain dish, and the salt remaining dissolved in boiling water. Both solutions are then acidified with hydrochloric acid, and the observation is made whether a strong turbidity or only an opalescence thereby ensues, as in the former case niobic and pelopic acids, and in the latter tantalic acid are to be suspected. A few drops of tincture of galls are then added to each, and while stirring, the color of the precipitate must be minutely noticed. Niobic acid, from the Bavarian Columbite, which contains pelopic acid, fused in the manner prescribed, with carbonate of potassa, and the resulting mass pulverized and treated with cold water, gives a solution in which, after it has been cleared and separated from the residue, hydrochloric acid produces a strong turbidity, and the tincture of galls, subsequently added, a dark orange-red precipitate. When the residual salt is dissolved in boiling water, and acidified with hydrochloric acid, a turbidity results, but tincture of galls produces only an orange-red precipitate indicative of pelopic acid.

When compounds have to be examined, which are free from tungstic, molybdic and silicic acids, for tantalic, niobic, and pelopic acids, the substance to be tested, already in a powdered condition, can be fused at once with carbonate of potassa in small portions in the loop of a platinum wire. The presence of titanitic acid does not interfere, because the titanate of potassa formed, only slightly passes into solution by the after treatment of the fused mass with water. Four parts of carbonate of potassa are mixed with the substance to be tested, and when the basic constituents are in a low degree of oxidation, one part of nitrate of potassa is added. After pulverization, the fused mass is first treated with cold, and then with boiling water as already described, and after the solutions have cleared, they are drawn off with a small siphon or pipette from the residue of basic constituents, which is, at last, mixed with insoluble titanate of potassa if titanitic acid were present. Each solution is now acidified with hydrochloric acid by itself in a porcelain dish, and a few drops of tincture of galls added. As before stated, a light yellow precipitate indicates tantalic acid, an orange-yellow pelopic acid, and a

dark orange-red niobic acid. When manganese is present, the fusion with carbonate of potassa converts it into manganate of potassa, which dissolves in cold water with a green color; but as this salt, by the addition of hydrochloric acid is first converted into permanganate of potassa, and subsequently into chloride of manganese, with which tincture of galls produces no precipitate, it is not prejudicial to the operation.

If a compound of tantalic, niobic, and pelopic acids contains silicious matter, the examination for these acids will answer without any alteration for minerals, which can be decomposed by hydrochloric acid, as Wöhlerite and Bukolite: but if not decomposable by that acid, it must be fused with soda and borax, the mass heated to dryness with hydrochloric acid, and afterwards treated with water. The acids remaining undissolved, are then washed on a filter with acidulated water, and after being dried and fused with five times their volume of carbonate of potassa in small portions on a platinum wire, the fused beads are pulverized and treated as above directed. The presence of silicic acid does not throughout prevent the reaction on the acids under consideration.

Both pelopic and niobic acids were formerly prepared by means of water from their chlorides, as other methods were not so advantageous.

But the separation of both was incomplete, and even when it was repeated in such a way that the chlorides obtained were decomposed into their corresponding acids by means of water, and these again converted into chlorides by treatment with charcoal and chlorine, the acids could not be obtained even moderately pure, and though the operation was repeated twenty or thirty times, the result was still unsuccessful.

After several laborious but vain researches, Rose succeeded in converting, under very peculiar conditions, a small quantity of niobic acid, which had been separated from pure niobate of soda, into a chloride. It was mixed with an extremely large quantity of charcoal, exposed to a strong current of chlorine, and subjected from the beginning to a very gentle temperature. This experiment led to the most surprising results. The pure niobic acid

gave, instead of the white chloride of niobium, the purest yellow pelopic acid, and this could always afterwards be obtained, but only by following strictly certain rules of manipulation and precaution. By the observation of a modified method, the white chloride could be obtained from the same acid.

From the preparation of both these chlorides, ROSE concludes that the same metal is contained in them as is contained in the acids prepared from them by means of water. But these acids once formed cannot be converted into one another like their corresponding chlorides, or only by a very circuitous process.

The quantity of oxygen has not, up to the present time, been determined directly, but the yellow chloride of pelopium contains more chlorine than the white chloride of niobium, hence pelopic acid must contain more oxygen than niobic.

Niobic acid cannot, however, be converted into pelopic, even by the most powerful oxidizing agents; neither does this result ensue by either direct or indirect oxidation. The behavior of the two acids before the Blowpipe also varies considerably. Such a relation is so peculiar, that there is no analogy to it in the whole domain of Chemistry.

It appears, nevertheless, that by some, but only few, reducing means, from the acid analogous to the yellow chloride, a little oxygen could be removed.

The proportion of oxygen in both acids, as deduced from that of the chlorine in the chloride, is a very anomalous one, and only in the two degrees of oxidation of sulphur, is a similar case found.

It is still doubtful whether a small proportion of oxygen is not contained in the carefully prepared white chloride, which may therefore be regarded as an acid salt; but the quantity of oxygen, according to the most exact investigation, is so extremely small that it is hoped the chloride will be obtained perfectly free from it. As, under all circumstances, pelopic and niobic acids are oxides of the same metal, these substances must only have one appellation. ROSE has selected that of niobium. The highest state of oxidation of this metal, formerly pelopic, must thus be called niobic acid.

§ 22. *TITANIUM*—Ti—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

It is found as an Acid $[\text{Ti O}^2]$ *per se*, and also in combination with bases, thus :—

a. Per se, in *Anatase*, *Arkansite*, *Brookite*, and *Rutile* ; the latter generally contains some Protoxides of Iron and Manganese ; —*Arkansite*, according to the examination of WHITNEY, is pure titanic acid, with only a trace of iron, and not a niobate, as inferred by SHEPARD, and has the crystalline form and specific gravity of *Brookite*. His trials make the specific gravity 4.085. Its insolubility in acids is strong presumptive proof that it is not titanic acid in combination with a base, since all the known titanates are soluble in acids ;

Brookite—*Prismatic Titanic Ore*—occurs in crystals of a hair-brown color, passing into deep orange-yellow, more or less translucent ; streak yellowish-white ; lustre brilliant, metallic adamantine. Insoluble and indecomposable in boiling hydrochloric acid, even when reduced to powder. Alone on charcoal it is infusible, but it is entirely soluble, and forms a brownish-yellow glass, with microcosmic salt. This species occurs with *Anatase* and *Crichtonite*, at Bourg d'Oisans, in Dauphiné, on the Tête-noire, in Savoy, and in large distinct crystals in Snowdon, in Wales. It is, however, a rare mineral ;

b. In combination with Earths and Metallic Oxides ; as with Lime and Silicate of Lime, in *Titanite* and *Sphene* ; with Lime, Protoxides of Cerium, Uranium, Iron, Manganese, Tin, *et cetera*, in *Pyrochlore* ; with Zirconia, Yttria, Oxides of Cerium, Iron, Calcium, and traces of Potassa, Magnesia, Silicic Acid, and Oxide of Tin, in *Polymignite* ; with Protoxide of Iron, in many rich slags, *exempli gratia*, in *Titaneisen*, *Crichtonite*, *Menaccanite*, *Nigrin*, *Iserine*, *Ilmenite*, *Volcanic Iron*, *Eisensand* ; and in all refractory Iron Ores whose slags are of a vitreous nature ;

c. As a very small ingredient in some minerals, it is found in *Cymophane*, *Kyanite*, *Achmite*, and in some varieties of *Mica*.—Titanium occurs likewise in *Perowskite*, *Polykras*, *Aeschnite*,

Euxenite, Yttrotitanite, Oerstedtite, Tschewknite, and Mosandrite.

When Titaniferous Iron Ores are smelted, the Titanium sometimes separates in the metallic state, and is observed upon the slags in small copper red crystals. It has been seen in beautiful perfect cubes upon a slag obtained from the smelting works near Frankfort-on-the-Maine.

Examination for Titanium.

The titanium contained in Anatase, Rutile, Titanite, and Sphene, can be detected by the behavior of these minerals with fluxes; *id est*, the first two comport themselves with borax, microcosmic salt, and soda, like titanous acid, the other two afford only the titaniferous color with microcosmic salt. In other minerals, where titanium forms an ingredient, it is somewhat difficult to detect it by the aid of the fluxes, as the other metallic oxides in combination hinder the reaction. If, however, ferruginous titanium ore be dissolved in microcosmic salt, and the glass, which exhibits the color of sesquioxide of iron only, treated for a long time in the reducing flame, a more or less brownish-red tinge is imparted to the assay, similar to that produced by ferruginous tungstic acid. The intrinsic quantity of titanium present, is deduced from the depth of color assumed by the glass. If the quantity present be great, the characteristic reaction of oxide of titanium—titanous oxide—will be obtained by treating with tin in microcosmic salt upon charcoal; but if only a small portion be present, this end is not attainable. Titanium, when forming an essential or appreciable ingredient in minerals, may be detected by a very simple method, as follows:—Fuse gradually the finely pulverized mineral in a platinum spoon, with from six to eight times its volume of bisulphate of potassa; dissolve out in hot water,—temperature about 200.8° Fahr. = 93.8° C.,—and allow the solution to subside; decant off the clear supernatant liquor into a flask containing more hot water, and boil. If the mineral contained even small quantities of titanium, it will be deposited as a white powder,—titanous acid,—provided iron is not present in the state

of protoxide, otherwise it will have a yellow tinge, probably from holding some of the iron in combination. This precipitate is to be separated by filtration, and examined, without beingedulcorated, with microcosmic salt, either on a platinum wire or charcoal. If the quantity be so small that no violet color is imparted to the microcosmic salt, the operator must add to the assay, treated on the wire, a small quantity of sesquioxide of iron, and when heated on charcoal, a piece of iron wire, and then heat the glass for some time in the reducing flame; it will appear, while hot yellow, and upon cooling red, analogous to oxide of nickel, or ferruginous tungstic acid. This reaction is produced immediately by the yellow precipitate, when heated with microcosmic salt in the reducing flame.

When such a precipitate is dissolved in borax, the iron has no effect upon the color produced by the titanic acid.

Should the operator not be perfectly convinced of the absence of tungstic acid, the method with bisulphate of potassa will be the most decisive for the detection of the titanium, as the titanic acid is precipitated on boiling the solution,—and any tungstic acid remains dissolved,—and can then be treated with microcosmic salt, as previously given.

If Polymignite be smelted with even ten times its volume of bisulphate of potassa, and the fused mass treated with warm water, little or no titanic acid is dissolved; it remains behind, with zirconia and traces of protoxide of iron, as a white voluminous mass, and can readily be recognized by collecting on a filter, and then treating some of the precipitate with microcosmic salt. The metallic titanium found in ferruginous slags dissolves with difficulty in borax, but is pretty readily soluble in microcosmic salt. The crystals which the German author applied in this research had been examined under the lens and considered as pure, but the yellow microcosmic salt glass was, in the reducing flame, neither *per se*, nor with the addition of tin, of a fine violet color, but somewhat reddish, like the ferruginous titanic acid.

§ 23. CHROMIUM—Cr—*Presence in the Mineral Kingdom.*

Chromium occurs only in an oxidized state in nature, thus :—

a. As Oxide [$\text{Cr}^2 \text{O}^3$] in a mechanical mixture with Quartz and various kinds of primitive rocks, in *Oxide of Chrome*, or *Chrome Ochre*; as Oxide, with Protoxide of Iron, Alumina, and Magnesia, in *Chromate of Iron—Chrome Iron Ore*— $[\text{Fe}^2 \text{O}^3] + \{\text{Cr}^2 \text{O}^3\}$;

b. As Chromic Acid [$\text{Cr} \text{O}^3$] with Lead, in *Chromate of Lead*, and with Lead and Oxide of Copper in *Vauquelinite*. In addition to these, Oxide of Chromium and Chromic Acid form an extraneous ingredient in many Silicious combinations, to which many of them owe their beautiful green or blood-red color; as the precious *Emerald*, the *Pyrope*, *et cetera*.

Traces of Chromic Acid are present in *Schiller Spar*, and in some varieties of *Serpentine*. Chromium is also met with in Ilmenite, Wolchonskoite, Uwarowite, Pyrosklerite, Miloschine, Spinele, Chrysoberyle, Rothbleierz, and Melanochroite.

Examination for Chromium.

The presence of chromium in most of the above minerals is readily detected by their behavior with borax and microcosmic salt before the Blowpipe, the bead being of a beautiful green when perfectly cold. This color is produced by minerals which contain neither lead nor copper, particularly after treatment in the reducing flame. When the minerals contain oxides of lead or of copper, *exempli gratia*, Chromate of Lead and Vauquelinite, the glass bead affords, in the reducing flame, a grey or red color, the peculiar green of the Chromium being completely destroyed; in consequence of this, attention should be paid, in the examination of such minerals, to those colors only which they produce in the oxidating flame.

Minerals which contain little chromium, and a large quantity of other metals that yield coloring oxides, and afford no satisfactory chrome reaction with borax and microcosmic salt, can be

examined for chromium in the following manner. A small fragment of the mineral is first powdered as fine as possible, the powder mixed with three times its volume of saltpetre, and the mixture fused with the aid of the Blowpipe, either on the platinum wire or in a small platinum spoon. Chromate of potassa is thus produced, and may be dissolved in water in a porcelain capsule, over the spirit-lamp. If the clear solution be poured off from the insoluble metallic oxides into another capsule, supersaturated with acetic acid, and a crystal of acetate of lead, then laid in it, the latter dissolves, and the chromic acid instantly combines with the oxide of lead, forming a beautiful orange-yellow powder, which precipitates, and, after filtration, yields with borax or microcosmic salt, in the oxidating flame, a beautiful green glass. Even small traces of chromium may be detected in this manner. If the substance examined by this method for chromium should contain sulphur or sulphuric acid,—which is not, however, present in the natural compound,—the chromate of lead will be contaminated with sulphate, and the yellow color will be lighter in proportion to the quantity of the sulphate present. Silicates that contain only a little chromium, and much iron or other metals yielding colored oxides, and which afford with fluxes only the peculiar tinge of iron or of the other metals, cannot be examined for chromium by the foregoing method, silicious combinations not being decomposed by saltpetre, but requiring a different treatment; such as the following:—The finely pulverized mineral is to be fused to a bead on charcoal, with from one to one and a half parts of soda, and half a part of borax; the bead pulverized, treated with hydrochloric acid, and evaporated to dryness. The resulting dry mass is to be dissolved in water, the silicic acid separated by filtration, a few drops of nitric acid added to the solution, in order to convert the protoxide of iron into the sesquioxide, and the oxides of chromium, iron, and alumina, *et cetera*, precipitated by ammonia. This precipitate, after being collected on a filter, must be fused with saltpetre; chromate of potassa is thus obtained, which may be decomposed by acetate of lead, in the manner already described.

The silicates colored blood-red by chromic acid, namely, the

Pyrope and Spinnelle, from Ceylon, possess the property of becoming black and opaque by simple heating, but during the refrigeration, they become, by transmitted light, gradually yellowish on chrome-green, then almost colorless; and when perfectly cold they re-exhibit their original color. Those silicious combinations which are colored red, both by chromium and iron, are also rendered opaque by calcination, but, on cooling, immediately recover their redness and transparency.

§ 24. *TELLURIUM*—Te—*Presence in the Mineral Kingdom.*

Tellurium occurs in nature only in the metallic state; thus,—

a. As *Native Tellurium* [Te], which contains a little Gold and Iron;

b. With Gold and Silver, in *Graphic Tellurium*, from Nagyag; with Lead, in *Black Tellurium*, mechanically mixed with Telluride of Gold, and sometimes Sulphides of Silver, Copper, and Antimony; with Bismuth, a little Sulphide of Bismuth, and Selenium, in *Telluric Bismuth*; with Silver, Lead, and Gold, in *Yellow Tellurium*; and in a compound of Bismuth, Lead, Copper, and a little Nickel, in the *Needle Ore* from Siberia.

Examination for Tellurium.

A substance may be examined for tellurium either on charcoal or in a glass tube, open at both ends. In the first case, a white sublimate, with a reddish border, is obtained by heating the mineral in either flame, and which disappears, with a beautiful bluish-green tinge, when the reducing flame is directed on it. When the mineral is rich in tellurium, it tinges the flame bluish, while subjected to the action of the reducing flame. If the assay, thus treated, gives off the odor of horseradish, it contains selenium. If the mineral contains lead or bismuth, and the operation be performed on charcoal, the pure sublimate of oxide of tellurium is never obtained when the blast is continued for a few seconds too long, but, on the contrary, will always be contaminated with oxide of lead or bismuth. It will, in consequence, be safer to

pulverize the substance, mix it with an equal bulk of glacial boracic acid, place it on a broad charcoal support, and treat it in the reducing flame. The oxides of lead and bismuth thus formed, notwithstanding the action of the reducing flame, will be dissolved by the boracic acid, and will not produce a sublimate; while the tellurium volatilizes, and alone coats the charcoal. Should the mineral, at the same time, contain a large quantity of selenium, a portion of it will also be deposited on the charcoal, and the tellurium cannot then be well recognized. In such a case, it is always better to perform the operation in a tube open at both ends.

If the operation is performed in a glass tube in a similar manner to Antimony, a strong white vapor is obtained from telluriferous minerals, which deposits as a pulverulent oxide on the glass, and, by heating, fuses on the same part of the tube on which it is deposited to clear colorless drops, which may be more closely examined with a lens. Should selenium be present, however, a trace of selenite of tellurium will be formed, which also melts into clear colorless drops, incapable of being distinguished from pure oxide of tellurium by the unaided eye.

When the telluriferous metal contains much lead, a sublimate is obtained, which appears grey close to the assay, and white at a distance from it. The white portion can be fused to colorless drops, and is oxide of tellurium; the grey, on the contrary, does not fuse like oxide of tellurium, but exhibits a somewhat changed appearance, and forms only a semifused greyish coating on the glass. According to BERZELIUS, this substance is tellurate of oxide of lead.

If bismuth be also present in the telluriferous metal, it remains behind, while the tellurium volatilizes and deposits in the tube. By continued treatment in the flame, it oxidizes, but the surface of the bead is coated with fused brown oxide of bismuth.

§ 25. *ARSENIC*—As—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Arsenic is found very abundantly in nature. It occurs :—

a. Metallic, either *per se*, or in combination with other metals ;

namely, as *Native Arsenic* [As], which generally contains traces of Antimony and Iron; with Cobalt, in *Speisscobalt*, which often contains traces of Iron, Copper, and Sulphur; with Nickel, in *Arsenical Nickel*—*Kupfernickel*—which sometimes contains traces of Iron, Lead, Cobalt, Antimony, and Sulphur; with a lesser proportion of Nickel, in *Weissnickelkies*, from Schneeberg; with Antimony, in *Arsenical Antimony*—*Arsenik-Speisglanz*;—with Bismuth, in *Bismuth Blende*; and with Silver, Iron, and Antimony, in *Arsenical Antimonial Silver*.

b. With Sulphur, both *per se* and combined with other Sulphides; namely, *per se*, as *Sulphide of Arsenic*, red [As S²]*—Realgar*; yellow [As S³]*—Orpiment*;—with Iron, in *Arsenical Pyrites*; with Cobalt and a little Iron, in *Bright White Cobalt*; with Nickel and Iron, in *Nickel Glanz*; with Copper, Antimony, Iron, and Silver, in *Grey Copper*—*Fahlerz*;—with Silver and a little Antimony, in the light red variety of *Ruby Silver*—*Argent Rouge Arsenic*, Necker;—with Nickel and Antimony, in *Antimonial Nickel*—*Nickelspeiss-glanzerz*;—and with Copper and Iron, in *Tennantite*.

c. As an Acid; thus,—as Arsenious Acid *per se* in *Oxide of Arsenic*—*Arsenikblüthe*—[As O³]; and as Arsenic Acid and Arsenious Acid, combined with other bodies; namely, as Arsenic Acid, with Lime and Water, in *Pharmacolite*; with Oxide of Cobalt and Water, in *Cobalt Bloom*; with oxide of Copper and Water, in *Condurrite*, *Euchroite*, *Erinite*, *Rhomboidal Arseniate*, and, accompanied by Alumina, in *Liroconite*; with Oxide of Nickel and Water, in *Nickel Ochre*, which often contains traces of Oxides of Cobalt and Iron, and Sulphuric Acid; with Protoxide of Iron and Water, in *Scorodite*; with Protoxide and Sesquioxide of Iron, and Water, in *Cube Ore*, or *Arseniate of Iron*, from Cornwall; with Oxide and Chloride of Lead, in *Arseniate of Lead*, from Johann Georgenstadt and Cornwall, which also contains traces of Iron and Phosphoric Acid; and with Oxide and Basic Sulphate of the Oxide of Iron, together with Constitutional Water, in *Pitchy Iron Ore*, or *Eisensinter*; lastly, as Arsenious Acid, combined with Oxide of Cobalt, in a pulverulent substance from Schneeberg.

As many of the above mentioned Arsenic combinations, partly *per se*, and partly in combination with other substances, are subjected, on a great scale, to operations for the reduction of the metals which they contain, and as Arsenic is a metal which is very difficultly separated in the dry way from a number of other metals, it must form more or less a constituent of the *Dressed Ores*, and of the various intermediate products of the smelting operation. To the latter belong especially the products which are obtained in Arsenic Smelting Works, as well as many of those which are obtained in Silver Works, as *Rohstein*, *Bleistein*, *Tutty*, *Abstrich*, *et cetera*, and also the *Speiss* produced in Cobalt Smelting Works, and often even the *Smalt*.

Examination for Arsenic.

The examination for arsenic is in most cases very simple. It should, however, be ascertained whether the arsenic exists in the compound in a metallic state, or as an acid. Native Arsenic is instantly recognized by the alliaceous odor which it exhales in volatilizing, when treated on charcoal before the Blowpipe. In a glass matrass, it sublimes in a metallic state, leaving a nonvolatile metallic mass behind, which, by fusion with lead and cupellation on bone ashes, sometimes yields a button of silver. The combinations of arsenic with sulphur, as Realgar and Orpiment, burn with a whitish-yellow flame, when ignited by the aid of the Blowpipe on charcoal, and give off a dense greyish-white vapor, which smells like garlic, and deposits in part on the charcoal. In a flask it fuses, boils, and sublimes. The sublimate is transparent, and of a dark-yellow or red color. Heated in an open glass tube, it burns and deposits white arsenic on the upper side of the tube; at the end of the tube, held upwards, the smell of sulphurous acid is readily recognized. Metallic arsenic is best obtained from sulphide of arsenic by the following method:—The compound is first decomposed in an open glass tube into arsenious and sulphurous acids, the former deposits in a crystalline form in the tube, and the latter escapes. For this purpose, the tube must be held obliquely, and heated a very little above the

specimen under examination, in order that the ascending vapor may pass over the hottest part of the tube, and be thus perfectly burned. The glass tube is then to be drawn out close to where the arsenious acid has collected, the latter forced into the part thus retracted, and reduced with charcoal, by the method which will be more fully treated of hereafter, for the reduction of pure arsenious acid to the metallic state. Most of the combinations of arsenic with other metals possess this property, that when a small portion of them is placed on charcoal and heated in the reducing flame, a part of the arsenic immediately volatilizes, and covers the charcoal with a white coating. When the quantity of arsenic present is considerable, a dense greyish-white smoke ascends from the assay, which diffuses itself, and may be readily recognized, even at a considerable distance, by its alliaceous smell; but if the quantity is inconsiderable, the above vapor is not always seen during the blowing, nor the arsenical smell experienced. In this case, the red hot assay should be brought close to the nose, in order that the arsenic escaping may be recognized by its peculiar smell. If a small quantity of arsenic is combined with a metal from which it is with difficulty separated, as with nickel, the compound should be melted with proof lead in the oxidating flame, and the smell observed, to ascertain if arsenic is volatilized.

Many compounds of arsenic with metals afford metallic arsenic when heated in a glass flask; instance some varieties of Speisscobalt, Weissnickelkies, Arsenical Antimony, *et cetera*; while others yield none, *exempli gratia*, Arsenical Nickel—Kupfernickel. All the metallic arsenides which occur in nature, when heated in an open glass tube, yield arsenious acid, which sublimes in combination with oxide of antimony, when the latter metal is present in the substance under examination.

If a metallic compound yields no arsenical smell when heated on charcoal *per se*, or with proof lead, and gives no sublimate of arsenious acid in an open tube, the presence of a minute trace of arsenic may still be detected by the following method:—

Friable metallic compounds, and such as can be reduced to powder, should be pulverized, and of the nonpulverizable, a por-

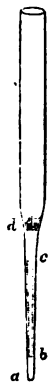
tion sufficient for an assay should be filed off. Artificial nickeliferous alloys, prepared on a large scale, do not require reduction to a finely divided state, as they are obtained either in a fine powder, or as a slightly adhering, easily pulverizable mass; melted nickel, on the other hand, German silver, and similar compositions, must be divided in this way. From 75 to 100 milligrammes of the metallic powder are then to be mixed in an agate mortar, with from five to six times as much saltpetre, and the mixture strongly ignited on a platinum spoon in the oxidating flame—see p. 81—by which means the metals are oxidized, and the arsenic acid formed is combined with the free potassa of the saltpetre. The fused mass is then removed, the spoon placed in a porcelain cup, water poured on it, and the whole warmed over the spirit-lamp, by which treatment the arsenic acid formed, the carbonate of potassa, and the remaining undecomposed nitrate of potassa are dissolved. If the metal to be examined contains zinc, lead, tin, or bismuth, slight traces of these metals will be present in the solution, as free potassa dissolves the oxides of these metals; these traces do not, however, hinder the recognition of the arsenic. The solution, which should be somewhat concentrated, is then diluted, without being separated from the residue, with spirit of wine, and powdered tartaric acid dissolved by a moderate heat in the alcoholic solution, after decantation from the residue, until it affords the acid reaction on litmus paper.

The bitartrate of potassa formed should then be allowed to deposit from the liquid in which it is insoluble. If the solution appears clear, it is to be carefully poured off from the precipitated salt into a porcelain basin, a small crystal of nitrate of silver placed in it, and the whole heated over the lamp. Should the solution contain arsenic acid, it combines with the oxide of silver, forming an insoluble arseniate of silver, which falls to the bottom as a brownish-red powder, but if the solution is free from arsenic, the nitrate of silver dissolves without producing any precipitate. Arsenical sulphides, when heated on charcoal in the reducing flame, readily yield a strong or a weak arsenical smell. If the quantity of arsenic contained in a sulphide be extremely minute, neither the vapor nor the smell of arsenic can be perceived.

Such substances,—to which belong *Rohstein*, *Bleistein*, *Flue-rakings*, *et cetera*,—must be pulverized, the powder mixed with soda, and the mixture fused on charcoal in the reducing flame. The sulphur is by this means separated, by combining with the radical of the soda, while the arsenic is volatilized, and may be recognized by its characteristic smell. The native arsenical sulphides, according to their various constituents, when heated in a bulb tube, sometimes yield a little sulphide of arsenic, with much metallic arsenic, as Arsenical Iron ; sometimes only sulphide of arsenic, for instance, Nickel Glance ; and sometimes no sublimate, *exempli gratia*, Bright White Cobalt. In a tube open at both ends, however, they all yield arsenious and sulphurous acids.

Pure arsenious acid may be tested before the Blowpipe, either on charcoal with soda, or by a method which *BERZELIUS* has given. The latter method is decidedly the safest, and most accurate when small quantities are to be examined. The following is the mode of procedure :—A glass tube is to be drawn out so that the diameter of the elongated part may be equal to that of a strong knitting-needle, and the end then sealed up. In order to detect the arse-

nious acid, which may be even less than one milligramme, it is to be introduced into the elongated part, forced into the extreme end—*a*, Fig. 45,—and a fragment of charcoal placed over it, about the size represented by *b c* ; the part of the tube occupied by the charcoal is then heated in the flame of the spirit-lamp, until the charcoal becomes ignited, and the part containing the arsenious acid gradually introduced into the flame. The arsenious acid is volatilized, and on coming in contact with the ignited charcoal, is reduced, forming in the cold wide part of the tube a deposit of metallic arsenic. When the quantity of arsenious acid under investigation is extremely minute, a black trace of metallic arsenic is only obtained between *c* and *d*. If the part of the tube drawn out be gradually



heated, nearer and nearer the black trace, the latter may be made to form a ring on the glass, and if the part retracted be cut off at *c*, and the part *d* held in the flame of the spirit-lamp, the arsenic may be volatilized, and the peculiar alliaceous smell recog-

nized. This method is far more delicate if cyanide of potassium be substituted for charcoal. In this case also, other compounds, besides arsenious acid, can be examined.

When the arsenic is present as an acid, combined with earths or metallic oxides, three modes can be employed for its detection :—

The first and simplest is, to mix the substance with soda, then treat it on charcoal in the reducing flame, and examine whether the smell of arsenic is evolved. This test is not, however, sufficiently delicate in all cases, especially when the arsenical acids are combined with metallic oxides, which are easily reduced, and form, with metallic arsenic, readily fusible arsenical alloys, which yield with difficulty the smell of arsenic. This is more particularly the case with oxide of nickel.

The second method for examining the compounds for arsenical acids, combined with earths, is to mix the substance with charcoal powder, and heat the mixture in a small tube having a bulb blown on one end of it, by which the arsenical acids are reduced, and metallic arsenic sublimed. Metallic arseniates and arsenites, with the exception of arsenite of copper,—known in commerce under the name of SCHEEL'S Green,—cannot be examined for arsenic in this way, because arsenical alloys are often formed by the bases accompanying the acid being reduced with it, from which arsenic cannot be sublimed.

To the compounds of the arsenical acids with earths, which admit of being examined in this way, belongs, in particular, arsenite of lime, which is often obtained only in exceedingly small quantities, in medico-chemical examinations for legal purposes. The subjoined method of procedure is advantageous in such a case :—The arsenite of lime is gently ignited, then mixed with fresh ignited charcoal, and introduced into the end of a sealed tube—Fig. 46—a part of the closed end of which is elongated, so that the mixture fills it up to *a*. The tube is next heated, gently at first, in order to drive off the moisture, and then the part *a*, containing the substance, heated in the Blowpipe flame until near the fusing point of the glass. The arsenic

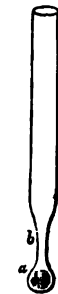


Fig. 46.

is by this means reduced, and collects in the narrow part *b* of the tube, where, from the very small surface over which it is spread, the most minute traces may be detected.

The third method for the detection of arsenic in arsenites and arseniates having earths or metallic oxides for their bases, is to mix the substance with three times its bulk of saltpetre, and proceed in the manner already described for the examination of arseniferous metals, in which the arsenic cannot be detected by treating on charcoal in the reducing flame.

§ 26. *SELENIUM—Se—Presence in the Mineral Kingdom.*

It occurs most frequently in the following minerals :—

With Lead, in *Selenide of Lead*, or *Selen Blei*, which often affords traces of Cobalt and Iron ; with Copper, in *Selenide of Copper* ; with Silver and Copper, with some earthly matters, in *Eukarite* ; with Lead and Copper, in *Selenide of Lead and Copper* ; with Lead and Cobalt, in *Selenide of Cobalt and Lead* ; with Lead and Mercury, in *Selenide of Lead and Mercury* ; and as a minute constituent in *Telluric Bismuth*, and in some varieties of *Swedish Lead Glances*. Selenide of Cobalt and Lead has much the aspect of Selenide of Lead. It gives off when heated in a closed tube a sublimation of Selenium, and exhibits with the fluxes the reaction of Cobalt by coloring them blue.

Examination for Selenium.

The examination for selenium is so simple, that even a minute trace of it in any metallic compound may be easily recognized by the following procedure :—

A small fragment of the compound is to be ignited on charcoal in the oxidating flame, and immediately held under the nose. If the substance contains selenium, a strong disagreeable smell is given off, similar to decaying horseradish, and which is peculiar to this metal. If much selenium is present, a reddish vapor is evolved before ignition commences, which consists of finely divided selenium ; but afterwards, a steel-grey metallic glistening subli-

mate is deposited on the charcoal, which, on the exterior edges, sometimes passes into violet, and in thin layers appears blue.

Selenium may also be readily separated from its compounds in its simple form, by merely roasting the assay in an open glass tube—see page 74—and, during the operation, inclining the tube, in order to oxidize the other elements, when this metal will sublime with a reddish color. If the quantity of selenium present be pretty large, the part of the sublimate nearest the assay appears almost of a steel-grey; small crystals of selenic acid are sometimes deposited before the red sublimate, but they disappear on the application of a very gentle heat. If sulphur be also present, which is the case with the seleniferous lead glances, sulphurous acid will be evolved, and may be recognized at the end of the tube either by the smell or by moistened litmus.

If selenium occurs with tellurium, *exempli gratia*, Telluric Bismuth from Tellemarken, which contains selenide of bismuth, and if the specimen be subjected to the experiment in a glass tube, oxide of tellurium is first given off, and, by continuing the blast, the selenium deposits as a red coating between the oxide of tellurium and the heated assay. As sulphide of arsenic, however, sometimes sublimes in an open tube, like selenium, this experiment alone does not suffice; the substance should, therefore, be treated on charcoal, by which means it can be instantly decided whether the sublimate is selenium or arsenic.

γ. EXAMINATION FOR NONMETALLIC BODIES AND ACIDS.

§ 1. OXYGEN—O—and HYDROGEN—H—in combination as WATER—H O—Presence in the Mineral Kingdom.

Under this head, no regard is paid to the Water occurring in nature, as Rain, Snow, Spring, Lake, and Sea Water, *et cetera*, but to the presence of Water in Minerals, of which it often forms an essential constituent. It occurs in large quantities in some minerals and hydrates, and may likewise be contained in a mechanical state.

It would be superfluous here to specify all those minerals which

contain Water, as this information may be found under each described metal.

Examination for Water.

This examination is very simple, and is to be performed according to the subjoined directions :—The substance is to be placed in a small perfectly dry glass flask,—see Fig. 8, page 15,—and then heated over a spirit-lamp. If it contains water mechanically retained or chemically combined, it will in the former instance be entirely eliminated, in the latter only partly. The aqueous vapor will deposit in drops, on the upper part of the vessel, where it can be readily seen. If a substance, insoluble in water, contain its water chemically combined, an evolution of vapor seldom occurs on the first application of the heat ; but if the vessel be introduced further into the flame, and strongly heated, the water will be liberated, as in the above case.

During the examination for water, the operator should observe all phenomena that take place ; because, by so doing, he may distinguish various substances which are analogous to others in their outward appearances, *exempli gratia*, many substances containing sulphuric, phosphoric, or hydrofluoric acids, give off water, when strongly heated, which reacts acid with litmus or Brazilwood paper : and sulphates often generate so much sulphurous acid, that it can be readily recognized by its characteristic odor.

Fluorides, when strongly heated, evolve hydrofluoric acid, which attacks the glass vessel,—this occurs with fluoride of cerium.

After the water has been expelled from the arseniates, arsenious acid may sublime, as with Scorodite.

An alkaline reaction with turmeric paper, or reddened litmus paper, indicates the presence of ammonia.

Decrepitation and change of color must also be carefully observed by the experimenter, in order to discriminate with certainty the substance under examination.

§ 2. *NITROGEN—N—and OXYGEN—O—in combination as NITRIC ACID—N O²—Presence in the Mineral Kingdom.*

This acid occurs in *Native Saltpetre*, which is generally con-

taminated with Sulphate, Nitrate, and Carbonate of Lime, and some Iron and Chloride of Calcium ; and in *Nitrate of Soda*, which generally contains seven to eight per cent. of Chloride of Sodium.

Examination for Nitric Acid.

The native salts which contain this acid are readily recognized by deflagrating when ignited upon charcoal.

The substances which are infusible, containing nitric acid, must be heated to redness, after being perfectly desiccated in a glass tube, by which means dark yellow fumes, nitrous gas, are eliminated, and may be recognized by their peculiar color and smell. If the nitric acid contained in a substance be small, and firmly united to a base, so that no characteristic fumes are obtained, a portion of the substance must be triturated in a mortar, with about an equal weight of bisulphate of potassa, and the mixture then treated as previously given. The free sulphuric acid combines with the base, liberating nitric acid, and the tube becomes filled with nitrous gas, the brownish-red or yellow color of which is best perceived by looking perpendicularly into the mouth of the tube.

§ 3. *SULPHUR—S—and SULPHURIC ACID—H O, S O^a—Presence in the Mineral Kingdom and in Furnace Products.*

Sulphur is found beautifully crystallized in nature, but is often contaminated with Bitumen, Silica, Lime, Iron, Carbon, Water, *et cetera*. It occurs most frequently in combination with Metals.

Sulphuric Acid is only met with in nature, in combination with Alkalies, Earths, and Metallic Oxides, which combinations have been previously given. All dressed ores, containing an admixture of metallic sulphides, which are not perfectly smelted before roasting, give, when remelted, metallic sulphides, which are called *Stein*,—and often a small portion of such metallic sulphides is mechanically contained in many products which ought to be perfectly free from sulphur. Sulphur also forms partly an essential, and partly a secondary ingredient in many furnace products. It

may be considered an essential ingredient, in *Rohstein*, *Bleistein*, *Kupferstein*, *Tutty*, *et cetera*, and a secondary one, in *Workable Lead*, *Schwartz Kupfer*, *Slags*, and other minerals.

Sulphuric Acid occurs in many roasted ores which previously contained Sulphur.

Examination for Sulphur, and Sulphuric Acid.

Native Sulphur is easily recognized by submitting it to the flame of a lamp upon charcoal. It burns with a fine bluish color, generating sulphurous acid.

Compounds with Sulphur may be detected by different methods, *videlicet*:—

a. Sometimes by heating the substance strongly in a glass tube. Some metallic sulphides possess a higher degree of sulphuration; thus, Iron Pyrites give off sulphur, which deposits in the upper part of the tube. If such a combination contains, at the same time, arsenic, as, Mispickel and Nickelglance, no yellow sublimate of sulphur will be obtained, but a yellowish-brown one, consisting of sulphide of arsenic;

b. By roasting the substance in an open glass tube. If only a small trace of sulphur is present, no sulphurous acid smell will be perceived, but its presence may be recognized by holding a strip of moistened litmus paper in the tube,—it will be immediately reddened;

c. By heating the substance in the oxidating flame upon charcoal. If an appreciable quantity of sulphur be present, the sulphurous acid smell will be immediately recognized;

d. The most accurate method, when no selenium is present, is, to melt the powdered assay,—those substances that cannot readily be reduced to powder may be applied in small particles,—with two parts soda, and one part borax, upon charcoal. In this treatment, sulphide of sodium is formed, which, when moistened and applied to a piece of bright silver, blackens it. The addition of the borax is to prevent the sulphide of sodium formed from being absorbed by the charcoal.

As the selenides behave exactly like sulphides towards metallic silver, the substance must previously be heated *per se* on charcoal,

in order to ascertain whether *the characteristic horse-radish odor* is exhaled. When the two occur together in a substance, the sulphur examination must be first performed in the open tube.

Sulphuric acid can be detected when in combination with alkalies, earths, or metallic oxides :—

¹ By melting the substance with silicate of soda in the reducing flame. The sulphuric acid at first combines with a portion of soda, which becomes reduced to sulphide of sodium, and the glass subsequently assumes a red or dark yellow color, according to the proportion of sulphuric acid present. If the quantity of sulphuric acid be very minute, the color appears only after the glass has perfectly cooled. The above method is only suitable for *colorless* sulphuric acid compounds : colored sulphates must be submitted to the following treatment :—

² Smelted with two parts of soda, and one of borax, on charcoal in the reducing flame, and the resulting mass moistened with water, and placed on silver ; a tarnishing of the metal will indicate the presence of sulphur.

§ 4. PHOSPHORIC ACID— $P O_5$ —*Presence in the Mineral Kingdom.*

This Acid is always met with in nature in a combined state, and is found in small quantity in numerous minerals. It is often overlooked, because traces of it are so difficult to detect in a satisfactory manner. The minerals in which it forms an ingredient have been mentioned under Lime, Alumina, Yttria, Copper, Uranium, Lead, Iron, Manganese, *et cetera*.

Examination for Phosphoric Acid.

If a substance contains more than from four to five per cent. of phosphoric acid, the annexed examination is the best. Dissolve some of the substance in Boracic acid upon charcoal in the oxidating flame, and then introduce into the molten bead a piece of fine iron wire, and expose the whole to a strong reducing flame. The iron oxidizes, at the expense of the phosphoric acid, and borate and phosphate of iron are produced,

the latter of which fuses in a strong heat. As the globule cools, a phosphorescence is generally remarked on various parts of the charcoal, which proceeds from the crystallization of the phosphide of iron. When perfectly cold, the glass must be removed from the charcoal, and broken into particles between folds of paper, upon an anvil. By this means the phosphide of iron will separate as a globular metallic button, and can be submitted to a further examination. It should be attractable by the magnet, brittle under the hammer, and of a ferruginous color when broken. If the substance contained only a small quantity of this acid, the globule containing the phosphide of iron will be sensibly ductile when struck. When very little or no phosphoric acid is present, on breaking the button the iron wire will be found intact, except where it protruded beyond the assay.

If the substance under examination contained other ingredients reducible by iron, and capable of melting with it to a globule, as sulphuric or arsenic acid, *et cetera*, it must be examined for these, before being subjected to the above treatment.

Phosphoric acid may be readily detected in minerals, if only three per cent. be present, in the following manner:—A splinter of the mineral is placed between the platinum points of the forceps, dipped into sulphuric acid, and then heated in a dark room with the apex of the blue flame, when the outer flame will be immediately colored bluish-green, of a perfectly different shade to that produced by boracic acid, baryta, or copper. Many phosphoric minerals, when treated *per se*, in the blue flame, color the outer flame green; *exempli gratia*, Wavellite.

When reactions are not obtained by the foregoing methods, it is better to have recourse to the moist way. The substance free from sulphuric acid and alumina must be fused in the oxidating flame, with four times its volume of soda, in the platinum spoon, and the residuum dissolved with water in a porcelain basin over a spirit-lamp, and then allowed to settle. Some of the clear supernatant liquor must then be decanted off, mixed with acetic acid, reheated over the lamp, and a small crystal of nitrate of silver immersed in the menstruum. If the solution contains only a trace of phosphoric acid, a lemon-yellow precipitate of

phosphate of silver subsides. Should the mixture not have been sufficiently heated, previous to the addition of the silver salt, a white precipitate of dipyrophosphate of silver will occur, which cannot be readily distinguished from other compounds.

If the substance contains alumina, as Wavellite, Amblygonite, Lazulite, *et cetera*, the phosphoric acid cannot be readily detected in the manner last mentioned, because on melting the substance with soda, phosphate of alumina combines with it, and this combination dissolves out on heating with water, and, on the addition of acetic acid, deposits. The subjoined method will detect a trace of phosphoric acid in an aluminous compound. Triturate the powdered substance in an agate mortar, with a mixture consisting of six parts soda and one and a half parts silica, then melt the whole mass in the oxidating flame upon charcoal. Treat the residuum in a porcelain basin with boiling water—the phosphate and excess of carbonate of soda is dissolved, leaving silicate of alumina and soda. If the substance contains only a little alumina, some silicate of soda will also dissolve, but this will have no detrimental influence on the detection of the phosphoric acid. The clear liquor is treated with acetic acid, *et cetera*, as recently given. If silicate of soda be contained in the solution, it precipitates on the addition of acetic acid, but will not in the slightest degree prevent the yellow reaction of the phosphoric acid with the silver salt.

When examining for phosphorus in metallic phosphides before the Blowpipe, the pulverized assay must be smelted with nitrate of potassa on the platinum wire, and the residuary mass treated with soda, similar to a substance containing phosphoric acid. The phosphorus and the metal oxidize, phosphate of potassa is formed, and finally, the metallic oxide separates.

§ 5. CHLORINE—Cl—*Presence in the Mineral Kingdom.*

Chlorine never occurs in a free state in nature, but in combination with other bodies.

The minerals containing Chlorine have been previously given under Potassa, Soda, Ammonia, Lime, Magnesia, Silver, Mercury, Copper, and Lead.

Examination for Chlorine.

Chlorine may be detected in metallic chlorides in the following manner. Dissolve oxide of copper in microcosmic salt, on the platinum wire, until a diaphanous bead is obtained in the oxidating flame. The substance under examination is now to be added, and heated. If chlorine is present, the assay will be surrounded by a beautiful blue colored flame, inclining to purple, which disappears after some time, but can be reproduced by adding a fresh supply of the sample. Chlorine, therefore, cannot be confounded with any of the acids which occur in the mineral kingdom, for none of them afford the foregoing reaction ; and even those that impart a color to the flame when mixed with a copper salt, give no tinge when operated upon in a cupriferous microcosmic glass. Annexed is a second method for the detection of chlorine in a compound soluble in water. A small quantity of sulphate of iron or copper must be dissolved, a few drops of the solution placed upon a bright piece of silver, and the metallic chloride added ; by this procedure, the silver will be blackened, provided chlorine is present. According to MERLET, when the chloride is insoluble in water, it must, previously to being added to the solution, be fused with soda on a platinum wire. When a small quantity of chlorine is present, the spot produced upon the silver will be greyish-black.

If the operator is not fortunate in obtaining a satisfactory result by the above methods, he must resort to the moist way, which will detect the *minutest* trace of chlorine. When the substance is insoluble, it must be fused with soda, as above stated ; when soluble, it is only requisite to dissolve it in *distilled water*, and to add a drop of solution of nitrate of silver. If there be only one part of chlorine in ten thousand parts of the solution, a milkiness ensues.

§ 6. BROMINE—Br—*Presence in the Mineral Kingdom.*

Bromine occurs as a small ingredient in nearly all *Salt Springs*, as Bromide of Sodium and Magnesium.

Examination for Bromine.

Metallic bromides give, with a cupreous microcosmic bead, and also with sulphate of copper on a silver plate, the same reactions as metallic chlorides ; but the blue color which the flame assumes does not incline to purple, but becomes green, especially on the edges. To distinguish the two with certainty, the substance under examination must be fused with bisulphate of potassa in a small glass flask, with a long neck, over the spirit-lamp. Bromine and sulphurous acid will be eliminated ; the former fills the vessel with a yellow gas, readily recognized by its characteristic odor, although sulphurous acid be present.

In examining for bromine in salt springs, a considerable quantity of the liquid must be evaporated, until nearly the whole of the chloride of sodium is separated. The mother liquor is then poured off, evaporated to dryness, and the residue examined with bisulphate of potassa in a glass flask. In this case, however, iodine is generally liberated, and therefore the pure yellow of bromine is not obtained, as it acquires a somewhat violet tinge, from the presence of iodine.

Small quantities of bromides may be easily detected by passing a stream of chlorine through a solution of the substance under examination, and agitating with ether. As soon as the liquids have separated, the solution of bromine in ether is withdrawn by means of a pipette, and shaken well with potassa, bromide of potassium and bromate of potassa being thus formed. On the solution of these two salts being evaporated to dryness, and fused in a platinum basin, the bromate is decomposed with evolution of oxygen, and the mass,—containing now only bromide—must be distilled with an excess of sulphuric acid and binoxide of manganese in a small retort, to which a glass condenser has been adapted. If bromine be present, brownish or yellowish-red vapors appear immediately, and impart an orange-yellow color to starch-paste in the receiver.

Bromides are decomposed by hypochlorites, and concentrated sulphuric and nitric acids, bromine being set free, which imparts a yellow or yellowish-red color to the liquid.

§ 7: IODINE—I—*Presence in the Mineral Kingdom.*

Iodine occurs in *Rock Salt*—also in *Salt Springs* and most mineral springs—in very small quantities, and generally in combination with *Sodium*. It has also been detected in a Mexican mineral, combined with Silver, and in a Silesian one, in combination with Zinc.

Its presence in the vegetal kingdom does not belong to this work ; but we may mention that it is likewise found, in combination with Sodium, in most kinds of *Seaweed, et cetera*. In some of these productions, such as the *Fucus Serratus* and *Fucus Digitatus*, it exists ready formed, and, according to Fyfe, may be separated by the action of water ; but in others it can only be detected after incineration.

Examination for Iodine.

The metallic iodides, when treated with cupriferos microcosmic salt, impart a beautiful and deep green tinge to the Blowpipe flame, which cannot be confounded with the color given when the bead, *per se*, is fused in either flame. Metallic iodides, melted with bisulphate of potassa in a glass tube, eliminate iodine vapor, which displays a beautiful purple color. Sulphurous acid is also set free.

Mineral waters can be examined for iodine in the same manner as for bromine ; the presence of the former is indicated by the violet colored vapor. If an appreciable quantity of bromine be present, the gas evolved will be of a darkish yellow color. A good test for Iodine is nitrate of silver. The iodide of silver is of a pale yellow color, and is *very difficultly* soluble in ammonia. The chloride dissolves in ammonia with great facility.

With chlorine, with concentrated sulphuric acid and binoxide of manganese, and also with nitric acid, iodides exhibit a behavior similar to that of bromides ; iodine is liberated, which,

according to its quantity is precipitated as a black powder, or remains in solution, imparting a brown color to the liquid. On heating this, the characteristic vapors of iodine are obtained.

Free iodine produces with starch-paste, a blue combination ; if it is in minute quantity, however, the result may be a violet or rose-red tinge.

Bromine and iodine may be separated by adding to the liquid containing them, a solution of a subsalt of copper—sulphate of copper to which sulphurous acid has been added, serves the purpose ; a dingy-white precipitate of subiodide of copper falls down, which may be filtered off, and the filtrate tested for bromine by adding strong sulphuric acid ; if the slightest coloration occurs, the presence of this element is indicated.

§ 8. *FLUORINE—Fl—Presence in the Mineral Kingdom.*

Fluorine is always found in combination. The minerals in which it forms an essential or secondary ingredient have been cited under Soda, Lithia, Lime, Magnesia, Alumina, Yttria, and Cerium. Traces of Fluorine have been found in the teeth, and also in the bones, of different animals.

Examination for Fluorine, or for Hydrofluoric Acid.

Hydrofluoric acid is not so readily eliminated by heat, from substances in which fluorine forms an essential ingredient,—*exempli gratia*, Fluor Spar, Topaz, Cryolite, *et cetera*,—as from those in which it sometimes occurs as a contingent one, as in Mica and Hornblende, when it is expelled generally as hydrofluosilicic acid.

If a small quantity of hydrofluoric acid be held in feeble combination, in a mineral containing water, it is necessary to heat it in a test tube, and apply a strip of moistened Brazilwood paper to its mouth. As the temperature augments, fluosilicic acid will be evolved, a silicious circle formed at a short distance from the

assay, and the Brazilwood paper tinged straw-yellow, by the hydrofluoric acid.

According to the experiments of BONSORF, Brazilwood paper is likewise colored straw-yellow by phosphoric and oxalic acids, but not by sulphuric, nitric, arsenic, or boracic acids. As phosphoric acid, however, is not volatile in such an examination, the tinge acquired by the paper will indicate the presence of fluorine. This test is so delicate, that it is only necessary, at times, to moisten the compound fluoride with hydrochloric acid, on a watch glass, and then to apply the reagent paper. When a substance under examination for fluorine produces no etching upon the glass, and affords no reaction with the Brazilwood paper, the following method must be adopted:—Mix the assay with metaphosphate of soda,—formed by heating the microcosmic salt to incipient redness,—and heat the mass in the end of an open glass tube, in such a position that there will be an access of hot air from the flame; by this procedure aqueous hydrofluoric acid is formed, which can be recognized by its smell, being more suffocating than chlorine, and also by the etching produced by the condensation of vapor in the tube. Moist Brazilwood paper, applied to the extremity of the tube, will be instantly colored.

The subjoined is MERLET's method for the detection of this acid:—Pulverize the substance for examination, then triturate it to an impalpable powder, and mix it with an equal part of bisulphate of potassa. Heat the mass gradually in a moderately wide dry test tube. The judicious application of heat must be strictly observed, for if the operator first heats the part of the tube where the assay rests, the whole may be lost, on account of the glass being shattered. The spirit flame must first be applied to the fore part of the tube, and then made to recede slowly, until it fuses the assay. After the mixture has been for some time kept in a molten state, the lamp must be withdrawn, and the part containing the assay severed with a file. The fore part of the tube must then be well washed, and afterwards dried with bibulous paper. Should the fluorine contained in the substance be significant, the glass tube, when held up to the light, will be

found to have lost its transparency, and to be very rough to the touch. Of all known substances, hydrofluoric acid is the most destructive to animal matter. When a drop of the concentrated acid comes in contact with the skin, instantaneous disorganization ensues, and a deep ulceration of a malignant character is produced. On this account, the greatest care is requisite in its preparation. It acts energetically on glass, the transparency of which is instantly destroyed, heat is evolved, the acid boils, and in a short time entirely disappears. A colorless gas, commonly known by the name of *hydrofluosilicic acid gas*, is the sole product. This compound is always formed when hydrofluoric acid comes in contact with a silicious substance.

§ 9. *CARBON—C—and CARBONIC ACID—C O₂—Presence in the Mineral Kingdom.*

Carbon occurs in nature in a pure state, as *Diamond*; and in combination with other bodies, in *Anthracite, Graphite, Stone Coal, Brown Coal, et cetera*.

Carbonic Acid occurs in minerals combined with bases which have been included in former sections of this work; it is also met with in the gaseous form, in mines and volcanic regions, and places where the earth contains large quantities of carbonate of lime; but as the examination of free gases does not come within the province of the Blowpipe, it is not requisite to enumerate the different places where this gas is exhaled.

Examination for Carbon and Carbonic Acid.

The diamond is so difficult to burn, that the ordinary Blowpipe is not sufficiently powerful. It may be heated to whiteness, in a covered crucible, without injury; but it begins to burn, in the open air, at about the melting point of silver—703·2° C.—and is entirely converted into carbonic acid gas. It is more quickly consumed in fused nitre, when the carbonic acid is retained by

the potassa : this is a simple mode of analyzing the diamond, by which it has been proved to be perfectly pure carbon. Some varieties of charcoal, when ignited with anhydrous alum in the oxidating flame, leave a recrementitious mass, consisting of ferruginous and earthy particles. When they are pulverized, mixed with nitrate of potassa, and heated strongly in a platinum spoon, they detonate, and afford carbonate of potassa.

The presence of carbonic acid is detected by the gas being eliminated with effervescence on heating a mineral containing it before the Blowpipe. It is also readily detected by moistening the substance with a few drops of hydrochloric acid—effervescence likewise ensues.

§ 10. *BORACIC ACID*— $B O^2$ —*Presence in the Mineral Kingdom.*

This Acid occurs combined with Water in *Native Boracic Acid* ; also in several minerals in combination with various bases. Those in which it forms an essential or secondary ingredient have been already adverted to, under Soda, Lime, Magnesia, and Alumina.

Examination for Boracic Acid.

According to BERZELIUS, Native Boracic Acid of Tuscany produces the following reactions :—Moistened with water and placed on Brazilwood paper, it bleaches the paper in about half an hour ; a solution in alcohol applied to curcuma paper imparts a brown tint.

Per se, on charcoal, it melts to a clear glass ; should it contain gypsum, the assay will be opaque when cold.

TURNER has recommended the following method for the detection of boracic acid in salts and minerals. Reduce the substance to an impalpable powder, and mix it with a flux consisting of four and a half parts of bisulphate of potassa, and one part of finely powdered fluor spar. Knead the whole into a paste, with a few drops of water ; then place the moist assay on the platinum

ring, and fuse the mass with the apex of the blue flame. During the fusion of the assay, fluoboric acid gas is liberated, which communicates a pure green color to the outer flame. Great care must be taken, when only a small quantity of boracic acid is present, for the coloring will be momentary. MERLET remarks that the reaction will be more certain, when three to four parts of the flux are used with one of the substance.

§ 11. *SILICIC ACID*— Si O^2 —*Presence in the Mineral Kingdom, and in Furnace Products.*

Silicic Acid forms a principal ingredient of the solid parts of our globe. It is found in nearly a pure state, in *Berg-Crystal, Quartz, Chalcedony, Hornstone, Cornelian, Flint, et cetera*. When any of the preceding minerals are colored, it is generally owing to the presence of Iron or Manganese. The native silicates have been cited under the Earths and Metals. Lastly, it is considered as an essential ingredient of dressed Ores and Slags.

Examination for Silicic Acid.

The Berg-Crystal and Milk-Quartz comport themselves with the fluxes, like pure silicic acid. The other varieties of Quartz, as regards their solubility in the fluxes, behave in the same manner; but the colors imparted to the borax or microcosmic glasses by the metallic oxides are more or less visible, according to the one contained.

The silicates are recognized by means of microcosmic salt and soda. They are decomposed in such a manner by the former, that their bases combine only with the free acid in the flux, and the silicic acid separates. This examination is generally performed on a platinum wire. The microcosmic salt must first be fused into a bead, the silicate added, and the whole treated in the oxidating flame. The glass bead, while hot, is clear, and the separated silica floats through it in a collected state. As glasses are obtained by numerous silicates, which are perfectly clear while

hot, but opaline on cooling, the operator must carefully observe the flux in its molten state, for the presence of silica. The silicious appearance is generally observable with those compounds of silicic acid which have lime, magnesia, glucina, yttria, or oxide of zinc, for the base.

Silicates of Zirconia, as Hyacinth and Zircon, are with great difficulty acted upon by microcosmic salt. The preferable method for the detection of the acid in these minerals has been given under Zirconia. If a substance contains a *small* quantity only of silicate, or be intermixed with some quartz, a glass is obtained, in which no skeleton of silica appears, owing to the solution of the silicic acid; it can be detected, however, by the aid of the moist way, proceeding in the manner given in the examination of the different Earthy Silicates. The silicic acid is separated in such a condition by this method, that it can afterwards be recognized with a little microcosmic salt on a platinum wire, or with soda on charcoal.

The silicates dissolve imperfectly in soda, on a platinum wire or charcoal, with effervescence. If it be a compound, in which the oxygen of the silicic acid is at least double that of the base, a clear glass will be obtained by the judicious application of soda, which will remain the same when cold, because the glass of silicate of soda acts as a solvent on the free bases.

A silicate containing an infusible base, gives a clear glass with *a little* soda. With a slight addition of soda, the glass is opaque; and with another supply, the assay becomes intact.

By the first addition of soda, a part of the base separates, which is redissolved, and the glass is clear; but when a large addition of the flux is supplied, so much of the base separates that the whole assay becomes infusible.

Substances in which only a small quantity of a silicate is contained, which is not at all detectible by soda, must be treated in the moist way. This method also must be resorted to in the examination of dressed ores, often containing metallic sulphides and arsenides, for silicic acid.

¶ *Examples of the Method of Procedure employed in Examining the different Compounds for all their constituents, with the aid of the Blowpipe.*

a. SALTS.

Salts come under the following heads :—

- a. Those which have Alkalies, for bases ;
- b. Those which have Earths ;
- c. Those which have Earths and Alkalies together ;
- d. Those which have Metallic Oxides ; and
- e. Those which have Metallic Oxides and Earths together.

These different salts may often be distinguished from each other by their external characters, and thus immediately recognized ; cases, however, occur, which cannot be so easily determined, and recourse therefore must be had to chemical analysis.

Such an examination can be easily performed with the Blowpipe, and the whole operation completed in a short time, if the proper method be undertaken ; which will be shown in succeeding examples.

1. *Sulphate of Potassa*— K O, S O_2 .

If this salt be in the state of powder, it is not possible to determine, from external appearances, whether the base is an earth, an alkali, or a metallic oxide ; the examination should therefore be conducted in the following manner :—A small portion of the salt is heated in a bulb tube, and examined in order to see if it yields water ; if it be employed in a crystalline state, it decrepitates, but yields none. The pulverized salt is next mixed with soda, this mixture fused on platinum foil in the oxidating flame, and examined during the fusion, in order to see if the whole mass fuses clear, or if any thing is precipitated. Sulphate of potassa fuses with soda on platinum foil to a perfectly clear glass ; the base, therefore, is an alkali. The particular alkali is determined by examining how the exterior flame is

colored, when a small fragment adhering to the moistened loop of the platinum wire is fused at the point of the blue flame. If the exterior flame exhibits a violet tinge, the presence of potassa is at once seen.

If the acid also is to be determined, a small portion must be treated on charcoal in the reducing flame. The salt under examination fuses, and is absorbed by the charcoal, which it coats with a white substance, formed of a number of orbicles, similar to that produced by sulphate of potassa, or a combination of potassium, with chlorine, bromine, or iodine. If the mass absorbed by the charcoal be moistened with a drop of water, it gives off a strong hepatic odor, and when removed from the charcoal and placed on a slip of silver foil, it produces a black spot of sulphide of silver on being moistened with water.

By this simple examination, it is ascertained that the constituents of this salt are sulphuric acid and potassa.

2. *Nitrate of Potassa—Saltpetre*— K O, N O_5 .

If the salt cannot be recognized by its external characters, the examination is conducted in the same manner as the preceding.

Heated in a bulb tube, it fuses readily, giving a clear glass, and yields no water. By this test it is already known that the base is an alkali; the test with soda on platinum foil may consequently be omitted.

Kept in a state of fusion on platinum wire, it colors the exterior flame violet. The base is consequently potassa.

Ignited on charcoal, its surface becomes exceedingly vesicular. The acid is therefore nitric acid.

3. *Iodide of Potassium*— K I .

When a small quantity is heated in a test tube with bisulphate of potassa or sulphuric acid, purple vapors are given off, which communicate a blue color to starch.

4. *Carbonate of Soda*— Na O, C O_2 .

In the examination of this salt, the procedure in the commencement is similar ; but it must be further treated, because the acid cannot be thus recognized.

The hydrated salt yields water in a bulb tube, which reacts neither acid nor alkaline.

It fuses to a clear glass with soda on charcoal. The base is consequently an alkali.

It fuses readily on platinum wire, and tinges the exterior flame strongly yellow. The base is consequently soda.

It melts on charcoal without detonating, is absorbed by the charcoal, but gives no sublimate. It is consequently not in combination with nitric, sulphuric, or hydrochloric acids.

It reacts alkaline with turmeric, or with reddened litmus paper.

It is now to be determined whether the soda is combined with carbonic acid. For this purpose, a small specimen of the salt should be fused with silicic acid on charcoal, and examined, in order to see if the two substances combine to form a clear glass with effervescence. This is the case with carbonate of soda, because its acid is evolved while the soda combines with the silicic acid. If the glass becomes of a yellow or a red color on cooling, the carbonate of soda is not free from an admixture of sulphuric acid.

5. *Chloride of Ammonium*— $\text{N H}^4 \text{ Cl}$.

In testing for water in a bulb tube, if the salt sublimes without leaving a residue, it must be an ammoniacal salt. If it then be tested with soda, for ammonia, and with a microcosmic salt bead saturated with oxide of copper, for chlorine, results are obtained, which can be obtained with chloride of ammonium only.

6. *Sulphate of Soda*— $\text{Na O, S O}_2 + 10 \text{ aq.}$

When heated in a test tube it gives off water ; the residue is

infusible. On charcoal, the flame is colored of an intense reddish-yellow. The residue gives off sulphide of hydrogen on being moistened with an acid.

7. *Potassa-Alum*— $\text{Al}^3 \text{O}^3, 3 \text{S O}^3 + \text{K O}, \text{S O}^3 + 24 \text{ aq.}$

When this salt is pulverized, it cannot be recognized as alum, unless from the taste; nor can we determine, from its external character, whether the base is an alkali or an earth, or whether both act as a base. The operation must, therefore, be conducted in a manner similar to that given for sulphate of potassa.

In a bulb tube, alum fuses, tumefies, and yields water, which reacts neither acid nor alkaline. The dry mass ignited, gives an acid, which reddens moistened litmus paper placed in the upper part of the tube. By the further examination of the salt, this acid is determined.

It forms an infusible compound with soda on platinum foil when its water is driven off. The base, therefore, appears to be an earth. But if a small portion be treated *per se* on a platinum wire at the point of the blue flame, the exterior flame will be colored deeply violet, from potassa. So far it is thus known that the salt has two bases, of which the earthy one remains to be ascertained.

As the salt appears perfectly white after the removal of its water, it should be tested with solution of cobalt; when it becomes colored of a beautiful blue, if, in separating the water from the substance on charcoal, a pure oxidating flame has been employed. This shows the base to be alumina.

The acid only now remains to be determined. It is first tested for sulphuric acid, because, in testing the salt in the bulb tube, litmus paper was reddened. When fused with soda on charcoal in the reducing flame, the fused mass, when laid on silver and moistened with water, produces a black spot of sulphide of silver. The acid is consequently sulphuric: separate tests for other acids produce no reaction; the salt, therefore, consists of sulphate of alumina and sulphate of potassa.

8. *Bromide of Sodium*—Na Br.

When heated in a test tube, with bisulphate of potassa, or strong sulphuric acid, this salt affords yellowish brown vapors of bromine.

9. *Sulphate of Magnesia*—Mg O, S O₃ + 7 aq.

The constituents of this salt are recognized in the same manner as those of alum.

In a bulb tube, it yields a large quantity of water, which does not react acid.

It is infusible with soda on platinum foil; the base, therefore, appears to be an alkaline earth.

The anhydrous salt, held with the forceps in the blue flame, tinges the outer flame, sometimes feebly, but evidently yellow, from an admixture of soda.

Solution of cobalt communicates to it a beautiful, but feeble rose tint. The base is consequently magnesia.

When the mass is fused with soda, on charcoal, in the reducing flame, it produces, when laid on silver foil and moistened with water, a black stain of sulphide of silver. It gives, with soda and silicic acid, the peculiar hepatic tint. The acid is, therefore, sulphuric, and the salt consists of sulphate of magnesia, which is sometimes contaminated with a little sulphate of soda.

10. *Chloride of Lithium*—Li Cl.

This salt imparts a reddish color to the Blowpipe flame.

11. *Celestine*.

Celestine decrepitates strongly, when heated in a bulb tube, but yields no water. The test with soda on platinum foil may be dispensed with, as this substance is instantly recognized as an earthy salt. It should be proved, however, whether it tinges the outer flame. A fragment held in the forceps, and treated with

the point of the blue flame, fuses, and communicates a reddish tint to the exterior flame. The color, which is much clearer than that given by a salt of lime, is similar to that produced by lithia, but not so beautiful. This shows the presence, therefore, of lithia or strontia.

A specimen is next treated with soda on charcoal, in order to ascertain if it is absorbed with the flux by the support. It fuses with it, the compound undergoes a strong ebullition, and sinks into the charcoal, as a strong hepatic mass. By this means it is at once determined, that the salt is either sulphate of baryta, or sulphate of strontia; but as it has been already found to tinge the outer flame red, and also that it is not very difficultly fusible, it may be considered as almost certain that it is not Heavy Spar, but Celestine.

If a specimen be ignited for a long time, on charcoal, in the reducing flame, the sulphide of strontium so formed treated with hydrochloric acid, and the resulting salt evaporated to dryness, and moistened with spirit,—as has been already more fully given under the head of Examination for Strontia,—the presence of strontia is recognized with certainty, by the red color of the alcoholic flame.

12. *Calcareous Spar.*

This salt is readily recognized from its external character. The method of procedure is similar to the foregoing.

Heated in a bulb tube it does not yield water.

Held in the forceps, and strongly heated in the apex of the blue flame, it does not melt, but appears luminous, and tinges the outer flame a feeble carmine-red. During the refrigeration, it generally becomes of a blackish-green color, which shows the presence of iron or manganese.

The ignited fragment, when moistened, and laid upon litmus paper, reacts alkaline.

From this behavior, the conclusion may be drawn, that the substance under examination is an alkaline earth, and this earth, lime, in combination with carbonic acid.

A fragment, next, should be dissolved on the platinum wire, with borax, to ascertain if it effervesces or not during solution, and to notice the color communicated to the borax glass. Calcareous Spar dissolves with effervescence, the carbonic acid escaping, and the glass has generally a feeble iron tinge. If a larger proportion be dissolved, the glass crystallizes during the refrigeration, presenting a number of sharp edges. By this means, it has now been discovered that the salt is a carbonate, containing a little oxide of iron, and that the base apparently is lime. A separate test for manganese determines whether the salt under investigation may not probably contain this metal also.

When a portion is treated with soda on charcoal, it is not dissolved; the soda is absorbed by the charcoal, and an infusible mass remains, which may be considered as lime.

13. *Pseudo Apatite.*

This mineral occasionally occurs in a locality not far from Freiberg, on the sides of small lodes, accompanying various silver ores, particularly dark Ruby Silver, and also with Iron Pyrites, in druses on gangues,—as an apparently changed natural product, of an hexagonal prismatic form, and a color partly yellowish-white, partly reddish-yellow; its fracture is almost earthy.

Many of the crystals, particularly the dark colored, when ignited in a bulb tube, decrepitate, and yield a small quantity of water only.

The fragments remaining in the bulb tube vitrify on the edges only, when intensely heated at the apex of the blue flame,—strongly phosphorescing at the same time, and when cold appearing of a lighter color. During the heating, the exterior flame is colored deeply yellow, from the presence of soda; with careful observation, however a dark green tinge may be evidently observed, shewing the presence of phosphoric acid. If the fragment held in the forceps be moistened with sulphuric acid, and then heated, the green tinge of the exterior flame becomes more decided, and notwithstanding the yellow color of the soda, may be recognized

as the reaction of phosphoric acid. It yields a regulus of phosphide of iron, with boracic acid and iron.

From the preceding investigation, it may be seen that the salt is a phosphate. Its behavior to vitreous fluxes and cobalt solution should now be examined, in order to be able to come to a conclusion as to the remaining constituents, particularly the bases.

This mineral dissolves slowly in borax forming a glass, which sometimes becomes streaked with opalescent lines, when a moderate quantity of the mineral is present ; with a still larger quantity, the glass becomes turbid during refrigeration. The reddish-yellow crystals give with borax a yellowish tinge, from oxide of iron.

It readily dissolves, with a slight effervescence, in microcosmic salt, and yields a glass colored by oxide of iron, which becomes turbidly streaked only when a large quantity of the substance is added ; when nearly saturated, it becomes, *per se*, during refrigeration, turbid, and exhibits imperfect facets. When perfectly saturated, an almost milk white bead is obtained.

With an equal weight of soda, the powdered mineral fuses on charcoal to a bead, which, under a continued blast, becomes infusible, and loses its orbicular form. A larger addition of soda is absorbed by the charcoal. If another portion of the powdered mineral be fused with soda on charcoal in the reducing flame, and the fused mass laid on a moistened silver plate, a black stain of sulphide of silver is produced.

When fused with soda and saltpetre on a platinum foil, it gives no reaction ; manganese is, therefore, absent.

Cobalt solution communicates a grey color to the powdered mineral.

From the behavior to borax, microcosmic salt, soda, and solution of cobalt, it can be seen that the mineral contains phosphoric, and also carbonic acid ; because it dissolves in microcosmic salt with effervescence ; and sulphuric acid, because, when fused with soda, it blackens silver. It may also be seen that the coloring ingredient is oxide of iron, and that the base is not alumina, as it gives a grey color, instead of a blue, with solution of cobalt.

If these Blowpipe reactions be compared with those of other combinations of phosphoric acid which occur in nature, it will be found that they resemble most those of *Apatite*.

We know, however, that apatite is a combination of phosphate of lime and chloride of calcium ; the latter being often replaced by fluoride of calcium. It is therefore necessary that the mineral should be directly examined for chlorine, hydrofluoric acid, lime, and other bases.

As we have already seen, under Examination for Chlorine, no certain reaction for this element can be produced in the dry way. If however, it be treated with nitric acid, it readily dissolves, with effervescence ; if this solution be diluted with distilled water, it will be rendered slightly turbid on the addition of nitrate of silver ; consequently, it is not perfectly free from chlorine.

When fused in a glass tube, closed at one end, with an equal quantity of bisulphate of potassa, faint marks are produced on the surface of the glass, at a small distance from the mixture, which remain when the closed end is cut off, the tube washed with water, and dried. The mineral must, therefore contain hydrofluoric acid.

The test for lime and other earths must be performed with the help of the moist way. It may, however, be considered nearly certain, that the principal constituent is lime, as the mineral forms during refrigeration, when fused with microcosmic salt, imperfect facets ; an admixture of other earths can only be detected, however, by recourse to the moist way.

For this purpose, a small portion must be treated with hydrochloric acid, and the earths precipitated as described under Lime, and more especially under Apatite. The mineral readily dissolves with the production of heat and evolution of gas,—with the escape of carbonic acid ; the solution, when diluted with water and spirit of wine, gives, with dilute sulphuric acid, an evident precipitate, which affords the reaction of gypsum before the Blowpipe, after beingedulcorated ; the solution, filtered from the gypsum, gives, with ammonia, a slight gelatinous precipitate, which, on further examination, behaves as ferruginous alumina.

The mineral contains, therefore,—

Phosphoric acid, a large quantity ;
 Carbonic acid, in smaller quantity ;
 Hydrofluoric acid, } in still smaller proportions ;
 Sulphuric acid, }
 Chlorine, very little ;
 Lime, a large quantity ;
 Alumina, in small quantity ;
 Soda, apparently only in small quantity ;
 Oxide of iron, as coloring matter ;
 Water, a little, probably only mechanical.

These constituents consequently show that the problematical mineral is a combination of phosphate of lime with phosphate of alumina, carbonate of lime, sulphate of lime, fluoride of calcium, and chloride of calcium.

14. *Chloride of Sodium*—Na Cl.

When heated in a glass tube, it decrepitates, giving traces of water, which were mechanically held between the layers of the crystals.

On platinum wire, it tinges the flame intensely yellow.

With a saturated copper bead of microcosmic salt, it colors the flame of a deep azure-blue, indicative of the presence of chlorine.

15. *Borax*—Na O, 2 B O³ + 10 aq.

It intumesces in the heat, giving off a large quantity of water.

When exposed on the platinum wire to the flame, it tumefies strongly, lastly giving a colorless bead. The flame is tinged yellow. Boracic acid is detected as follows :—Mix borax, sulphuric acid, and alcohol together, and ignite. A green flame indicates the presence of boracic acid.

16. *Phosphate of Lead*.

This mineral is readily distinguished, when in the solid state, by its external characters. In this case it is only necessary to subject it to direct examination for its known constituents. If it

be in the state of powder, however, it may be easily confounded with other minerals of the same color, and it should, therefore, be examined in the following manner :—

It should be first tested on charcoal in both flames, and its fusibility, and other characters from which a conclusion may be drawn, examined. The powder fuses to a bead, on charcoal, in the oxidating flame, which crystallizes during refrigeration, and becomes of a dark color. If any substance is observed to volatilize during the fusion, and deposit in part on the charcoal as a white sublimate, which admits of being driven further from the assay on the application of either flame, antimony is probably present. A separate test for antimony need not, however, be undertaken on this account, as other compounds may be present which would yield a white sublimate, and which may be accidentally discovered during the course of investigation. If the bead be treated long enough with a strong blast in the reducing flame, the charcoal will be seen to be yellow, as when coated with oxide of lead ; and if the blast be discontinued, the bead crystallizes almost instantaneously, during the refrigeration ; large facets of an almost white color, similar in brilliancy to mother-of-pearl, being formed. As this appearance is peculiar to phosphate of lead, and as a sublimate of lead was already obtained on the charcoal, it is not necessary to test for the presence of phosphoric acid and lead. It may however be done, in order to be perfectly convinced.

A globule of phosphide of iron and metallic lead may be readily obtained with boracic acid and iron, and a button of lead with soda on charcoal, which may be tested for silver by cupellation on bone ashes.

The appearance of a white sublimate when the mineral is treated *per se* on charcoal, is readily explained by the fact, that the natural combinations of phosphoric acid with oxide of lead always contain chloride of lead, which volatilizes. A separate test for chlorine, with a microcosmic salt bead saturated with oxide of copper, shows very clearly that the mineral contains chlorine.

17. *Nickel Ochre.*

This mineral may be easily determined to be a metallic salt. Its constituents are also readily recognized during the course of the examination.

It yields some water when heated *per se* in a bulb tube, its color becoming darker. Its behavior *per se* on charcoal is then to be examined. It sometimes diffuses an evident alliaceous odor when treated in the oxidating flame, and by treatment in the reducing flame it fuses, with the escape of some arsenic, to a black brittle metallic button, which is readily attracted by the magnet.

From this property, and from the mineral possessing a green color, it may be considered with a degree of certainty that it principally consists of arseniate of oxide of nickel.

But as natural arseniate of nickel is not always free from oxide of cobalt, oxide of iron, and sulphuric acid, it should be tested with borax, and a separate examination made for sulphuric acid.

It dissolves readily in borax, and yields a glass which is violet while hot, and reddish-brown when cold—oxide of nickel. If this glass be treated on charcoal in the reducing flame until all the nickel is precipitated, the glass will appear perfectly colorless when the mineral is free from cobalt and iron; if not free from these, it will appear blue from cobalt, or green from iron. The blue glass may, however, contain iron also; it is, therefore, necessary to fuse it, after being freed from reduced metallic particles, in the loop of a platinum wire; treat it for some time in the oxidating flame, and carefully observe whether it appears green or blue while hot; if the former is the case, it is not free from iron.

It is exceedingly difficult to separate completely, particles of reduced nickel from a glass from which the nickel has been thrown down in the metallic state; this is, however, easily effected if the precipitated nickel be melted with a particle of lead, and the glass treated in the reducing flame only. The nickel thus combines with the lead, forming a readily fusible alloy, from which the glass may be removed while in the molten state.

The examination for sulphuric acid is made by fusion with

soda and borax on charcoal in the reducing flame, and the fused assay treated on a moist piece of silver.

18. *Wolfram.*

The behavior of this mineral before the Blowpipe, as well *per se* on charcoal, as with fluxes, must be first determined, in order to be able to ascertain the principal constituents, unless the mineral is recognized from its physical character as wolfram.

It decrepitates when heated in a bulb tube, and gives very little water.

Its fusibility must then be examined. This operation is most conveniently performed in the forceps, in order to be able to observe, at the same time, whether the exterior flame is colored. It fuses with difficulty to a bead, on charcoal, without producing a sublimate; the surface of the bead presents small eminences, from a number of laminated crystals of a considerable size.

The mineral should now be tested with borax and microcosmic salt.

Borax dissolves it in the oxidating flame, without difficulty, to a clear glass, which is reddish-yellow while hot, when the quantity present is extremely minute, and pure yellow during the refrigeration, as when colored with oxide of iron; from a larger quantity, it appears blood-red while hot, and reddish-yellow on refrigeration. Treated for a few moments in the reducing flame, the tint becomes lighter, showing the color to be derived from oxide of iron only.

It is readily dissolved by microcosmic salt in the oxidating flame, to a clear glass, which appears reddish-yellow while hot, and somewhat lighter during the refrigeration; its appearance, however, shows only the presence of oxide of iron. If the glass be treated in the reducing flame, it becomes dark red; even when the quantity present is moderate, it becomes opaque. If a small quantity of the saturated glass be treated with tin on charcoal, for a few moments, in the reducing flame, it will assume a green color when cold. If such a glass be exposed for some time to a strong blast in the reducing flame, the green color dis-

appears, and a slight reddish-yellow tint remains behind, which undergoes no further change.

From these reactions with borax and microcosmic salt, we may conclude that, as the borax glass assumes a redder tint after being treated with the oxidating flame, and a more or less yellow color, when treated for a short time in the reducing flame, the mineral must contain manganese as well as iron; and as the microcosmic salt bead becomes dark red, when treated in the reducing flame, and assumes a green color on the addition of tin, which becomes reddish-yellow when exposed for some time to the reducing flame, tungsten must be present, as well as iron and manganese.

If the powdered mineral be fused with soda in a platinum spoon, the fused mass dissolved in water, and hydrochloric acid added to the clear solution, a white precipitate is produced, which, on heating the whole, becomes of a beautiful lemon-yellow color, and may be recognized as tungstic acid.

The constituents of Wolfram therefore, are tungstic acid, protoxides of iron and manganese, and a little water. The color of the mineral shows that the iron and manganese are present as protoxides, and not as sesquioxides.

β. ALUMINATES.

As the number of aluminates which occur in nature is very limited, and we have already treated of the most of them, when speaking of the examination for Magnesia and Alumina, a single example will suffice.

Spinelle, red, from Ceylon.

The first thing to be done, is to ascertain its behavior before the Blowpipe, both *per se*, and with fluxes.

Heated strongly in the apex of the blue flame while held in the forceps, it loses its form, but communicates no color to the exterior flame. If the thoroughly ignited fragment be viewed with reflected sun light, it appears almost black and opaque,

while hot, but becomes chrome-green during refrigeration, then almost colorless, and when perfectly cold it assumes its red color, unaltered. This change of color shows the presence of chromium.

With borax it dissolves with difficulty, even in a powdered state, to a clear, slightly yellowish, green colored glass, which does not become turbidly streaked.

It is dissolved by microcosmic salt with difficulty when in fragments, but fuses readily and perfectly, when it is employed in the state of a powder, to a clear glass, which has a reddish tint while hot, but during the refrigeration it becomes evidently, though slightly, of a chrome-green.

It is not dissolved by soda, but fuses to a vesicular mass without depositing a sublimate on the charcoal. Fused with soda on platinum foil, the presence of traces of manganese may be detected.

Solution of cobalt communicates a blue color to the fine powder.

From this behavior, we may conclude that, as the mineral dissolves with difficulty in borax, without producing a glass streaked with turbid lines; as by solution in microcosmic salt no skeleton of silicic acid remains behind; as it does not dissolve when treated with soda, no sublimate being at the same time produced; and as the powder receives a blue color from cobalt solution—the mineral under examination is an aluminate, not a silicate, which contains neither oxide of lead nor zinc.

As the aluminates readily admit of decomposition, by fusion with soda and borax on charcoal,—as has been mentioned when speaking of the examination of such combinations for Magnesia,—the alumina and magnesia, and an unimportant constituent, with the silicic acid, may be recognized with certainty in the spinelle from Ceylon, by further treatment of the fused mass.

γ. SILICATES.

The Silicates can be divided into—

- a. Monobasic; and,
- b. Polybasic Silicates.

The former may be readily recognized as silicates before the Blowpipe ; and the base, as it consists of one earth or of one metallic oxide, may often be recognized at the same time.

The latter can also be recognized as silicates by their behavior before the Blowpipe ; but the bases do not always admit of being so readily recognized.

In cases where the bases do not admit of being ascertained with accuracy during reactions before the Blowpipe, recourse must be had to the moist way, and the compound decomposed either by fusion with bisulphate of potassa in a platinum spoon, or by fusion with soda and borax on charcoal. The first method answers very well with monobasic silicates, silicates of alumina excepted ; the second may be employed with more advantage with polybasic silicates.

1. *Tabular Spar.*

Heated on a bulb tube, it undergoes no change ; it yields, however, a little water.

Held in the forceps, it communicates no tinge to the exterior flame, and fuses on the edges only to a semi-clear glass.

It is readily dissolved in large quantities to a clear glass, which does not become streaked with turbid lines.

It is dissolved by microcosmic salt to a clear glass, leaving a silicious skeleton behind. This glass when moderately saturated, becomes, during refrigeration, opalescent.

With an equal weight of soda, the powder fuses to a vesicular glass, which, on the addition of more soda, tumefies and becomes infusible.

Solution of nitrate of cobalt renders it difficultly fusible, producing a blue color on the fused edges only.

From these results, we may come to the conclusion that Tabular Spar, as it leaves behind a skeleton of silica when treated with microcosmic salt, is a silicate ; and that the base, as the mineral readily dissolves in borax, and as solution of cobalt communicates a blue color to it only where it fuses, must be lime.

To prove this fact more fully, it is only necessary to fuse the powdered silicate with bisulphate of potassa, and treat the fused mass in the manner already described under Lime.

2. Soap Stone.

Heated *per se* in a bulb tube, this mineral yields a little water, gives off an empyreumatic odor, and becomes black.

The mineral, when held in the forceps and heated in the oxidating flame, becomes white, contracts slightly, and fuses on the thinnest edges to a white enamel. It does not tinge the exterior flame.

Borax readily dissolves it to a clear glass, which generally possesses a slight ferruginous tinge.

Microcosmic salt decomposes it, leaving a residue of silica. The clear colorless glass, thus produced, crystallizes on cooling.

With a certain quantity of soda, it fuses to a clear glass, which becomes turbid when either too large or too small a portion of the flux is employed.

Cobalt solution communicates to it a reddish color, when it is exposed for a sufficient time in the state of powder to the oxidating flame.

From these reactions it is quite apparent that Soap Stone is a silicate of magnesia.

As this mineral, however, sometimes contains a little alumina, which cannot be detected by the aid of the dry way alone, the finely pulverized soap stone should be fused with bisulphate of potassa, in order to be certain that alumina is present, and the fused mass decomposed in the manner given under the head of Examination for Magnesia.

3. Pyrosmalite.

Heated *per se*, in a bulb tube, Pyrosmalite undergoes no change, but yields a little water.

In the forceps it fuses on the edges only, to a black metallic shining slag, which becomes red in the reducing flame.

It is dissolved by borax in the oxidating flame, with a slight effervescence, to a clear glass, of an amethystine color. This color disappears in the reducing flame, and the glass, unless saturated, becomes colorless.

If is with difficulty decomposed by microcosmic salt. When treated in the oxidating flame, it affords an amethyst colored glass, which contains a skeleton of silica ; it loses the color in the reducing flame.

With a little soda, it fuses to a black glass ; if the flux be increased, a difficultly fusible black slag is formed.

Pyrosmalite is therefore a silicate, the base of which is oxide of manganese. But as it dissolves with effervescence in borax, carbonic acid is present, combined with the oxide.

4. *Felspar.*

Per se, in a bulb tube, it undergoes no change, and when perfectly transparent, yields no water.

In the forceps it is very difficultly fusible, melting only on the edges to a semi-clear vesicular glass. Like soda, it tinges the exterior flame yellow.

It is dissolved to a clear glass by borax, very slowly, and without effervescence.

It is perfectly decomposed by microcosmic salt, only when in the state of powder ; a skeleton of silica being left behind. The glass becomes opalescent on cooling.

The powder fuses with soda very slowly, and with effervescence, producing a very difficultly fusible clear glass, perfectly free from vesicles.

Solution of cobalt communicates a blue color to the fine powder, on the fused edges only.

Felspar is thus a silicate ; and as it is very difficultly soluble in borax, coloring the exterior flame yellow, the base must consist principally of alumina, with a little soda ; but it cannot be determined, by the Blowpipe alone, whether or not this silicate is combined with small quantities of other silicates.

About 75 milligrammes of the finely powdered mineral should

therefore be melted with soda and borax, on charcoal, to a clear bead, which should be pulverized, the powder treated with hydrochloric acid, and the solution evaporated to dryness ; the chloride formed, dissolved in water ; the clear solution separated by filtration from the insoluble silicic acid, and the filtrate examined for the different earths, in the manner given under the Examination for Baryta, Lime, and Alumina. In this way it will be found, that the solution contains a large quantity of alumina, with a trace of lime.

The constituents thus found show that the silicate must be a felspar ; but whether it contains soda, or potassa with an accidental trace of soda, can be ascertained only by making a separate test for potassa, in the way given at page 103. Potassa-felspar appears to contain a trace of soda, as it generally colors the exterior flame more or less yellow. If a considerable precipitate of chloride of platinum and potassium is formed, in an alcoholic solution of the alkaline portions of the mineral, it is a potassa-felspar, which contains only a trace of soda ; but if no precipitate is obtained, it is a soda-felspar. A portion of the hydrochloric acid solution, prepared to examine for the earths, may be employed in testing for potassa, so as to avoid a second fusion of the substance with soda. An experiment may also be made in order to ascertain if fluorine is present.

5. *Rohslag, from the Freyberg Smelting Works.*

Per se, in the forceps, it readily fuses, retaining its black-green color, and tinges the exterior flame slightly yellow, from traces of soda.

It dissolves easily in borax to a clear glass, which is strongly colored by iron.

Microcosmic salt dissolves it to a clear glass, which is likewise tinged yellow, leaving behind a silicious skeleton.

It readily dissolves with soda on charcoal, with effervescence, to a black bead, which deposits no sublimate on the charcoal when

treated in the reducing flame, but frequently produces, after some time, a black spot on moistened metallic silver.

It gives the reaction of manganese, when fused on platinum foil, with soda and saltpetre.

From these Blowpipe reactions, *Rohslag* would appear to be a silicate, the principal base of which is protoxide of iron. But as it is known that such a slag also contains different earths, which cannot be detected with the Blowpipe, about one hundred milligrammes, in a pulverized state, should be taken, fused with soda and borax on charcoal in the oxidating flame, and the fused bead decomposed in the manner given under Baryta. Sulphuric acid, or bisulphate of potassa, produces an exceedingly small precipitate of sulphate of baryta, in the diluted solution of the chlorides. When the protoxide of iron is converted, by the addition of a few drops of nitric acid, into the sesquioxide, and a little chloride of ammonium added to the solution filtered from the baryta precipitate, ammonia throws down sesquioxide of iron and alumina: oxalate of ammonia produces, in the solution filtered from these substances, a slight precipitate of oxalate of lime: and, lastly, microcosmic salt gives a slight precipitate, in the solution filtered from the oxalate of lime, of magnesia, and protoxide of manganese, combined with phosphoric acid and ammonia.

If these different precipitates, after edulcoration, be treated before the Blowpipe, in the manner given already in speaking of the earths, it will be found that *Rohslag* consists of—

Silica,
Protoxide of Iron,
Alumina,
Lime,
Magnesia,
Baryta, with traces of
Manganese, Sulphur, and Soda.

As the fragment of slag employed was perfectly vitreous, and free from admixed particles of *Rohstein*, the minute quantities of sulphur it contains may be considered as combined with baryta or lime.

6. *Black Copper Slag, from the Freyberg Smelting Works.*

Held in the forceps, it fuses readily, but does not color the exterior flame ; when moistened with hydrochloric acid, however, it colors the exterior flame bluish-green, from the formed chloride of copper.

It behaves to borax and microcosmic salt like *Rohslag*, but the beads appear, with the same quantity of substance, more intensely colored, and the microcosmic salt does not contain so large a skeleton of silica. The iron present is therefore greater, and the silicic acid less.

With soda it fuses on charcoal, with effervescence, to a black bead. If this be treated for some time in the reducing flame, a white sublimate of oxide of lead is deposited on the charcoal.

By a reduction test with a large quantity of soda, metallic particles are obtained, which behave to boracic acid like a mixture of lead and copper.

It shows the presence of a slight trace of manganese, when fused with saltpetre and soda on platinum foil.

It may be seen from these reactions, that *Black Copper Slag* is apparently a silicate of protoxide of iron, containing slight traces of oxides of lead and copper, and protoxide of manganese. The presence of earthy bases must be ascertained by the moist way.

For this purpose, about one hundred milligrammes of the finely pulverized slag should be fused to a bead with soda, borax, and a button of pure silver, of about eighty milligrammes in weight, in the reducing flame, and the melted bead further treated in the way described for analyzing the *Rohslag*. In this way, it will be found that *Black Copper Slag* contains alumina and a trace of lime, and both must be considered as bases.

If the silver globule, free from slag, be treated with microcosmic salt for a short time on charcoal in the oxidating flame, and the resulting bead re-melted with a little tin in the reducing flame, it will become brownish-red and opaque during refrigeration, from the presence of protoxide of copper.

Black Copper Slag therefore consists of—

Silica,
Protoxide of Iron,
Alumina,
Lime, with traces of
Oxides of Lead and Copper, with Protoxide
of Manganese.

8. COMPOUNDS OF METALLIC OXIDES.

The compounds of metallic oxides occurring in nature, which cannot be considered as Salts, are either oxides or hydrates. Some of these exist *per se*, while others are in combination with different simple minerals. Those which can be submitted to ignition in a bulb tube without yielding water, are oxides; and those which yield water, are either hydrates, or oxides containing hydrates.

Metallit oxides occurring in the products of smelting works are often contaminated with sulphuric acid, arsenical acids, antimonious acid, and a portion of the oxides combined with these acids; but they never contain chemically combined water.

The method of procedure, in the investigation of such compounds of metallic oxides, may be gathered from the two following examples.

1. *Brown Earthy Cobalt.*

Ignited in a bulb tube, it yields water of an empyreumatic odor, which does not affect the color of litmus paper.

It suffers no change on charcoal, in the oxidating flame; in the reducing flame, it gives off a slight alliaceous odor, but does not melt or yield a sublimate.

Borax and microcosmic salt dissolve it, in the oxidating flame, to a clear dark violet glass, which appears, after treatment in the reducing flame, greenish while hot, and pure blue when cold.

Soda does not dissolve it. It yields a mass, colored deep green,

from the presence of manganese, when they are fused together on platinum foil in the oxidating flame.

When reduced with soda, and the resulting mass levigated, a metallic powder is obtained, which is attracted by the magnet, and which, when dissolved in microcosmic salt, appears green while hot, and blue when cold—cobalt and iron.

If a borax bead, saturated with this mineral, be treated for some time with a little lead on charcoal in the reducing flame, the lead will congeal, on cooling, with a clear surface; consequently no nickel is present.

From these reactions, we may conclude,—as the brown earthy cobalt gives no water in a bulb tube, dissolves in borax and microcosmic salt, in the oxidating flame, with a dark violet color, which changes in the reducing flame to blue, and produces, with soda on platinum foil, a green color,—that it consists of hydrates of the oxides of cobalt and manganese: and, as it gives off a slight odor of arsenic, when heated on charcoal, and, as the borax bead exhibits, when treated for a short time in the reducing flame, by which the manganese is reduced to the state of protoxide, a green color while hot, and only blue when cold, that it must be contaminated with arsenic acid and oxide of iron.

2. *Abstrich, from the Freyberg Smelting Works.*

It undergoes no change when heated in a bulb tube.

In a tube open at both ends, it behaves itself similarly.

It readily fuses on charcoal, spreads itself, and is reduced with effervescence to a very fluid metallic button, which gives off a strong alliaceous odor when kept in a state of fusion in the oxidating flame, coats the charcoal with a sublimate of oxide of antimony and oxide of lead, and finally behaves itself like pure oxide of lead.

Borax readily dissolves it in the oxidating flame, to a clear green glass, which retains its color when cold. In the reducing flame, on charcoal, the bead spreads, and a number of lead globules are reduced, which have a strong alliaceous odor, and which coat the charcoal with oxide of antimony and lead.

If the small lead globules be combined and removed from the glass, and the latter again fused to a bead by a continued application of the reducing flame, it becomes colorless, and remains so when cold. But if a fragment of *Abstrich* be reduced in the reducing flame on charcoal, with a borax bead of half its size, the reduced lead removed, and the borax glass melted to a bead, it will appear greenish, and, when further treated with tin, of a pure vitriol-green, from iron.

Microcosmic salt dissolves it also, in the oxidating flame, to a clear green glass, which retains its color when cold. The color of the glass undergoes no change on charcoal in the reducing flame while hot, but during the refrigeration it becomes turbid, and of a greenish-yellow tint. If this glass be treated for a short time with tin, it becomes during refrigeration, of a perfect blackish-grey color, from reduced antimony ; if, however, it be kept long enough fluid in the reducing flame, the antimony will be volatilized, and a glass obtained, which becomes perfectly red during the refrigeration, from protoxide of copper.

It is instantly reduced with soda to a grey, somewhat brittle, metallic button.

From these reactions, it may be seen that *Abstrich* is an oxide of lead, containing a little oxide of copper, and a still smaller quantity of oxide of iron : and that a portion of the oxide of lead is combined with an arsenical and antimonial acid, as no volatile body is set free when heated in an open tube.

c. SULPHIDES, ARSENIDES, AND SELENIDES.

The system to be followed in these researches, is similar to the one given under Metallic Oxides ; but in many cases, where numerous metals are present, producing with the fluxes a confused coloration, different steps are to be taken. In most instances, before the fluxes are applied, the assay should be freed from sulphur and arsenic, by roasting it upon charcoal. The following examples may probably be of great service to the experimentalist.

1. *Zinc Blende, from Przibram.*

When heated in a matrass, it falls to powder, and when this powder is heated to redness, no volatile ingredients are eliminated. If heat be applied to the powder in an open vessel, its color changes to a lighter hue, and sulphurous acid gas is evolved, which reddens moistened litmus paper.

On charcoal, in the reducing flame, the color of the powder likewise assumes a lighter tinge, and traces of sulphurous acid are evolved. In the reducing flame, *sinteration* ensues, and the charcoal becomes coated with a yellowish sublimate, which is white when cold. When treated with a solution of cobalt, it assumes a beautiful green color—oxide of zinc. The fine powder dissolves copiously in borax and microcosmic salt; if it be heated with these fluxes on charcoal, in the oxidating flame, the glass obtained is clear, and presents the ferruginous tint only; but when the glass is over saturated, it appears turbidly streaked.

The roasted, as well as the unroasted powder, dissolves with effervescence in soda on charcoal, forming an opaline mass. After continued blowing with the reducing flame, the charcoal is at first covered with a reddish-brown sublimate, the color of which is most distinct on refrigeration, and may be immediately recognized as oxide of cadmium; a large sublimate of zinc then occurs, and the soda is imbibed by the charcoal. If the unroasted substance is operated upon, a strong hepatic smell results, when the spot where the soda has been absorbed is moistened with water.

From the preceding reactions, we may infer that the ingredients of Zinc Blende are—

Zinc, and
Sulphur, with portions of
Cadmium, and Iron.

2. *Cupriferous Sulphide of Bismuth.*

It decrepitates slightly, and ultimately melts when heated in a flask, but volatile ingredients are not evolved.

When heated in an open glass vessel, sulphur sublimes, and in the under part of the tube a deposit appears, which, when strongly heated, melts to brownish drops ; and the substance, after cooling, is yellowish by transmitted light—oxide of bismuth.

It melts readily upon charcoal, with ebullition and spirting. When the blowing is uninterrupted, nearly the whole volatilizes, and the charcoal presents an orange-yellow coating. This deposit becomes lemon-yellow on cooling, and appears to be pure oxide of bismuth. If the residuary matter be melted with borax in the oxidating flame, a beautiful green glass is obtained, which will remain unaltered when cold—iron and oxide of copper. On treating this bead with tin, the assay will be of an opalescent red color, when cold—suboxide of copper.

If the roasted mineral is melted on charcoal, with bisulphate of potassa, in the oxidating flame, and the fused assay be treated according to the method given under the examination for Bismuth, the operator will not find lead. When the mineral is melted with borax, on charcoal in the reducing flame, a greenish glass is obtained, which acquires a vitriol-green tinge when treated with tin—protoxide of iron. When a small portion of the mineral is purified with proof lead, on charcoal, and submitted to the oxidating flame till the sulphur is eliminated, then melted with a little boracic acid, a button of copper remains behind, which forms about five per cent. of the quantity taken. The mineral, therefore, consists of—

Bismuth,
Sulphur,
Copper, and a trace of
Iron.

3. *Bournomite, from Kupriaz, near Freyberg.*

When heated *per se* in a flask, it decrepitates, giving off volatile ingredients.

Heated in an open vessel, it evolves a large portion of sulphurous acid, and also a dense white vapor, which deposits partly on the upper, and partly on the lower side of the flask. The upper

portion is volatile,—oxide of antimony,—while the under is not, and being present in large quantities, appears to consist of antimonide of lead.

It melts readily on charcoal, coating it with oxide of antimony, which congeals to a black bead, possessing a rough surface. By continued blowing, the bead re-melts, and the charcoal becomes thickly coated with oxide of lead. If the globule, greatly reduced in volume, be treated with borax in the reducing flame, a colorless glass is formed,—no iron present,—but when subjected to the oxidating flame, a red cupriforous glass is obtained,—suboxide of copper,—and a cupreous button, which is brittle, from a small trace of sulphur, but when treated with a little soda, it becomes perfectly malleable.

The cupreous globule, when cupellated with lead, gives a button of silver.

This Bournonite, therefore, is composed of

Sulphur,
Antimony,
Lead,
Copper,
Silver—0.1 per cent.

4. *Nickel-Glance.*

This mineral decrepitates when heated *per se* in a flask, evolving a large quantity of sulphide of arsenic ; as the heat augments, it melts. When heated in an open glass tube, arsenious and sulphurous acids are eliminated.

Heated on charcoal, a dense arsenical vapor is evolved, part of which coats the charcoal, and is readily expelled. If the greater part of the arsenic be volatilized, the assay fuses readily to a globule, quite brittle on cooling. If this be melted with borax on charcoal in the reducing flame, the glass appears, after perfect refrigeration, greenish-blue. When the glass is separated from the metal, and heated on the ring of the platinum wire in the oxidating flame, it appears green while hot, from cobalt and oxide of iron, and after cooling only slightly blue, from oxide of cobalt.

On charcoal with tin, the glass assumes a vitriol-green color, mingled with much blue. The metallic globule, separated from the borax glass, and treated again in the oxidating flame with borax on charcoal, gives the tinge from oxide of nickel only ; the nickel separates in the metallic state, in the reducing flame, and the glass appears colorless.

From the above reactions, we may conclude that Nickel Glance contains—

Arsenic,
Nickel,
Sulphur,
Iron, and
Cobalt.

5. *Grey Copper, from Freyberg.*

It decrepitates in a flask, and gives, if melted, a red sublimate of sulphide of arsenic.

The powdered mineral fumes pretty strongly, in an open glass tube. This vapor deposits a white sublimate on the glass, and appears to be a mixture of arsenious acid and oxide of antimony. At the upper end of the tube, a strong smell of sulphurous acid will be recognized.

The powder melts readily, *per se*, on charcoal, to a globule which fumes strongly. The vapor deposits partly on the charcoal, and consists of oxide of antimony. If a sufficiency has been applied, a second sublimate is obtained, nearer to the assay than the sublimate of antimony ; while hot it is yellowish, on cooling white, and assumes a green color with a solution of cobalt,—oxide of zinc.

On account of the large quantity of sulphur present, no arsenical smell is perceived when the assay is treated *per se* on charcoal ; but if another portion of the mineral be treated with soda on charcoal, the sulphur separates, the arsenic volatilizes, and is distinctly perceptible from its alliaceous smell. If a portion of the mineral be gradually roasted on charcoal, and treated with borax on the same in the reducing flame, a bottle-green glass is

obtained, which becomes vitriol-green after dressing with tin,—iron,—and leaves a somewhat liquid metallic globule, which has a light copper color, and therefore appears to consist of copper and a little antimony. If this globule be treated for some time with borax in the oxidating flame, the glass will be tinged red, from suboxide of copper, and the globule assumes a pure copper color. If the cupriferous button be cupellated with lead, a small silver globule is procured.

Grey Copper, therefore, consists of—

Sulphur,
Antimony,
Arsenic,
Copper,
Silver,
Iron, and
Zinc.

6. *Rohstein, from the Freyberg Furnaces.*

Heated strongly in a matrass, it gives off no volatile particles, but assumes a blackish color.

Heated in an open glass tube, it evolves sulphurous acid, recognizable by moistened litmus paper, or by its odor.

On the lower side of the tube, near the assay, a thin white coating is formed, not volatile, and appearing to be antimonious acid.

It melts very readily *per se* on charcoal; and, after a continued exposure to the reducing flame, coats the support with three different metallic oxides:—The sublimate which is at first produced, is deposited at some distance from the assay; it is white, and admits of being driven from one side to the other with the oxidating flame—oxide of antimony. That produced later, is immediately in contact with the antimonial sublimate; it is, while hot, of a lemon-yellow, and on cooling, of a sulphur-yellow color—oxide of lead. The third deposit, which is near the assay, appears only in very thin white laminæ; if it be moistened with a solution of cobalt, and heated strongly, but carefully,

in the oxidating flame, it becomes green—oxide of zinc. *Rohstein* emits arsenical vapor, when heated with soda on charcoal.

When this substance, in a finely powdered state, is roasted carefully on charcoal, and a part of the roasted assay dissolved in borax on a platinum wire in the oxidating flame, a clear glass is obtained, which presents the color of oxide of iron only. If this glass be treated on charcoal with tin for a short time, it becomes reddish on cooling, from copper; after a longer reducing flame, the copper is separated, the glass remains clear when cold, and of a pure vitriol-green hue, from the presence of iron.

If another portion of the roasted *Rohstein* be dressed with soda, borax, and lead, and heated on charcoal in the reducing flame, those metallic oxides are reduced, combining with the lead.

The plumbiferous alloy, while yet in a molten state in the Blow-pipe flame, evolves antimony; when the blowing is discontinued, it cools with a clear surface—no nickel. If the plumbiferous globule be melted with boracic acid till the greater part of the lead is separated, and it be then melted with microcosmic salt on charcoal in the oxidating flame, and the resulting glass bead treated with tin, a very distinct reaction of copper is also produced.

The constituents of this *Rohstein* are, therefore,—

Sulphur,
Iron,
Antimony,
Lead,
Copper,
Zinc,
Arsenic, and—according to a quantitative silver
assay,—also,
Silver—0.18 per cent.

7. *Lead Speiss, from the Freyberg Furnaces.*

Heated to redness in a glass tube, it liquefies, forming a black mass, but evolving nothing volatile.

A little arsenious acid is eliminated in an open glass tube ; near the assay it exhibits a white nonvolatile deposit, probably of antimonious acid, and on the upper part of the glass tube, a smell of sulphurous acid is distinctly observed.

On charcoal *per se*, in the reducing flame, it melts at the commencement to a globule, and evolves arsenical vapor ; but after continued blowing, a crust forms on the surface and the globule becomes infusible. But if another splinter of the mineral be melted with twice its volume of lead and boracic acid on charcoal in the reducing flame, a sublimate of oxide of antimony sufficiently distinct is obtained, and a dense arsenical vapor is observed.

If a part of this Lead Speiss in a finely powdered state, be roasted on charcoal, by which means the sulphur and a part of the arsenic volatilizes,—but the other part of the arsenic is converted into arsenic acid, and, without being capable of separation, combines with the metallic oxides formed,—and the roasted assay melted with borax on charcoal in the reducing flame, a black opaque glass and a ramous white metallic globule are obtained ; a portion of the glass diluted with borax on charcoal, and treated for some time in the oxidating flame, exhibits a greenish-blue color ; melted in the ring of a platinum wire, and treated in the oxidating flame, it assumes a reddish-yellow color, which on refrigeration becomes yellowish-green ; it is therefore colored by iron and cobalt. If the metallic globule be melted on charcoal with microcosmic salt in the oxidating flame, a green glass results, colored by nickel and oxide of copper, which, when treated with tin, is, on refrigeration, red and opaque, owing to the presence of suboxide of copper.

According to the above reactions, Lead Speiss consists of—

Arsenic,
Sulphur,
Nickel,
Iron,
Cobalt,
Antimony,
Copper ;—and gives, on cupellation,
Silver,—0.048 per cent.

If convenient, to ascertain the presence of nickel, iron, and cobalt, with greater certainty, and to observe the colors of the metallic oxides separately in the fluxes, a rather circuitous method, similar to the following must be pursued.

Roast about seventy-five milligrammes of finely divided Lead Speiss, on charcoal in the reducing flame, to volatilize the sulphur and the other metals, so as to form arseniates of nickel and cobalt.

Melt the roasted assay with equal parts of soda and borax, on charcoal in the reducing flame. By this treatment, nickel, copper, and the greater portion of the cobalt, antimony, and arsenic are reduced, forming a limpid metallic globule, while the iron, as protoxide, and the irreducible oxide of cobalt, remain dissolved in the flame. If the glass flows readily, and is free from metallic particles, the blast must be discontinued, and the arsenical metals separated from the glass.

Pound the glass, mix it with twice its volume of soda, and expose the assay on charcoal to the reducing flame. Then sift the grey metallic powder, attractable by the magnet, from the slaggy matters, dissolve it in borax, and either the color from iron, or that from iron with a little cobalt, will be the result.

When the metallic particles are treated with borax on charcoal for a long time in the oxidating flame, and a portion of the resulting glass bead dressed with more borax and some lead, and then exposed on charcoal to the reducing flame, the characteristic cobalt-blue tinge will be communicated to the flux.

The assay, freed from cobalt by the assaying with borax, must be again treated with this flux on charcoal in the oxidating flame, and the glass bead, while hot, separated from the metallic assay. It exhibits the color from nickel only. When placed on another part of the charcoal, and treated in the reducing flame, the nickel is separated in a metallic state, the glass assumes, on cooling, a brown-red color, and is opaque from suboxide of copper. After long blowing, the copper is also separated in a metallic state, and the glass has a tinge only of blue, from a trace of remaining cobalt. The copper does not appear to be reduced before the nickel is separated. If the borax glass, perfectly free from metallic

globules, be melted on charcoal with microcosmic salt in the oxidating flame, a glass darkly colored is attained : if it is melted in a similar manner on a platinum wire in the oxidating flame, it is infusible, and appears colored beautifully green by oxide of nickel and copper.

8. *Cupriferos Selenide of Lead—Selenide of Copper and Lead.*

This mineral decrepitates strongly when heated in a flask, but otherwise does not change. It gives a sublimate in an open glass tube, which, at the greatest distance from the assay, is red, and nearer to it steel-grey—selenium. No sulphurous acid is detectible, either by the smell of the upper end of the tube, or by moistened litmus paper. It also fumes on charcoal, smells strongly of selenium, melts on the surface only, and coats the charcoal at the commencement with selenium, which appears grey and of a shining metallic lustre, and afterwards also with oxide of lead. After continued blowing, a black slaggy mass remains, which melts readily to a globule, and imparts a bottle-green tinge to the glass, owing to the presence of an inconsiderable quantity of iron. The separated metallic globule is somewhat malleable ; *per se* on charcoal, it produces a strong sublimate of oxide of lead, and if treated with boracic acid on charcoal, gives a pure cupriferos globule.

The constituents therefore are :—

Selenium,
Lead,
Copper, and
Iron—a trace.

§. COMBINATIONS OF METALS WHICH CONTAIN LITTLE OR
NO ARSENIC.

The system to be followed in such researches is very simple, as may be deduced from the following examples.

1. *Native Amalgam.*

Heated strongly *per se* in a matrass, metallic orbicles deposit in its neck, which cohere on shaking the vessel. If the porous residue be first treated in the oxidating flame, and then in the reducing flame on charcoal with borax, the operator will obtain a colorless glass, and a metallic globule, which has the appearance of silver, and remains unchanged by cupellation. It is composed of—

Silver, and
Mercury.

2. *Workable Lead from the Freyberg Furnaces.*

If convinced that mercury is absent, a glass alembic is not necessary. It can also be dispensed with in the case of *Workable Lead*.

In the open glass tube it melts to a bead, which is coated with oxide, and gives nothing volatile. It melts readily on charcoal; has a moderately strong smell of arsenic; at the commencement it coats the charcoal with oxide of antimony; and also, after continued blowing, with oxide of lead.

Melted with borax on charcoal, in the reducing flame, a clear colorless glass is obtained, remaining so when melted in the ring of a platinum wire, and treated for some time in the oxidating flame; it is, therefore, free from iron and cobalt. Melted with soda and borax, on charcoal in the reducing flame, and the glass placed on a moistened silver plate, a black stain of sulphide of silver is often produced; it is, therefore, not always free from sulphur.

Treated with boracic acid on charcoal in the reducing flame, till only a small metallic particle remains, and this globule melted with microcosmic salt again on charcoal, in the oxidating flame, a greenish glass is obtained, which, if treated with tin, appears on cooling opaque-red, from copper. If the metallic globule be cupellated, after melting with microcosmic salt, a globule of silver remains behind.

According to the foregoing reactions, Workable Lead consists of,—

Lead,
Silver,
Copper—traces,—
Arsenic,
Antimony, and sometimes traces of
Sulphur.

3. *Impure Tin.*

On charcoal in the oxidating flame it behaves, as regards its easy oxidation, like pure tin. In the reducing flame, it gives a coating of oxide of tin, mixed with a dark yellow powder, which exhibits a lemon-yellow color on cooling, and appears therefore to be oxide of bismuth. As a confirmation, another part of this tin was melted with microcosmic salt on charcoal, first in the oxidating flame, and then in the reducing flame. The glass bead was, while hot transparent, but on cooling, became black and opaque, which indicated the presence of bismuth. Melted with borax, on charcoal in the reducing flame, a glass was produced, which was colored pure vitriol-green, by protoxide of iron.

The tin was contaminated with bismuth and iron.

4. *Black Copper, very Impure.*

Heated strongly in the open glass tube, it eliminates a little sulphurous acid, recognized by the introduction of moistened litmus paper. At some distance from the assay, a very slight white deposit is formed, having the appearance of oxide of antimony. On charcoal, *per se*, it melts with difficulty, yielding a sublimate of oxide of lead only. Melted together with proof lead and boric acid, in such a manner that one side remains free, while the lead dissolves, a distinct sublimate of oxide of antimony is produced. The remaining part of the metallic globule, which is free from lead, has a greyish-white color, and is scopiform.

The black copper, melted with borax on charcoal, in the reducing flame, produces a glass colored blue by cobalt, which

melts on a platinum wire in the oxidating flame, appearing, while hot, green, and on cooling, blue—cobalt and oxide of iron. The metallic globule remaining, after treatment with boracic acid, produces, with microcosmic salt, on charcoal in the oxidating flame, a glass, which appears of a beautiful green, both when hot and cold, and becomes opaquish-red with tin—nickel and copper. The remaining undissolved metallic globule still appears greyish-white, and is extremely dendritic. This appearance indicates the presence of arsenic, which is combined with nickel, and cannot be separated from this metal, either by boracic acid or microcosmic salt. A separate examination for arsenic, according to page 235, indicates the presence of this metal.

This *Black Copper* consists of—

Lead,
Copper,
Nickel,
Cobalt,
Iron,
Antimony,
Arsenic,
Sulphur; and, according to a separate examination, a little
Silver.

5. *German Silver, or White Copper—Argentan.*

No change ensues, when it is heated in a glass tube.

Melted on charcoal, in the reducing flame, it affords a sublimate, which is yellow while hot, and white when cold; with solution of cobalt, it assumes a beautiful green color, and therefore may be regarded as oxide of zinc.

The globule, melted *per se*, on charcoal, and treated with borax in the outer flame, till those metallic oxides, irreducible in the inner flame, are dissolved, should be remelted in the reducing flame to separate the metals. The glass thus obtained is blue, and does not change its color when fused on a platinum wire in the oxidating flame. Cobalt only is therefore dissolved.

The metallic globule, freed from cobalt and melted with microcosmic salt, on charcoal, in the oxidating flame, gives a glass colored dark green. A portion of this vitreous mass, treated with more microcosmic salt, on platinum wire, in the oxidating flame, affords a bead which, on cooling, remains of a beautiful green—copper and nickel. This bead, on being removed, and treated on charcoal with tin, becomes opaque-red on cooling, owing to the presence of copper.

The globule remaining undissolved, after treatment with microcosmic salt, is perfectly malleable, appears reddish-white, and consists, as it indicates a trace of silver only on cupellation, of copper and nickel.

The composition of this alloy, is, therefore—

Copper, with a trace of
Silver,
Nickel, with some
Cobalt, and
Zinc.

SECTION III.

SECTION III.

DESCRIPTION OF A CONVENIENT AND USEFUL BLOWING APPARATUS,

For Quantitative Analysis with the Blowpipe.

REPEATED quantitative examinations with the Blowpipe would weary even the most experienced operator, and it has, therefore, been found necessary to construct a blowing apparatus, which, provided the manipulator is always employed at the same place, completely remedies the evils attendant upon the use of the more common instrument.

It should possess the following qualifications :—

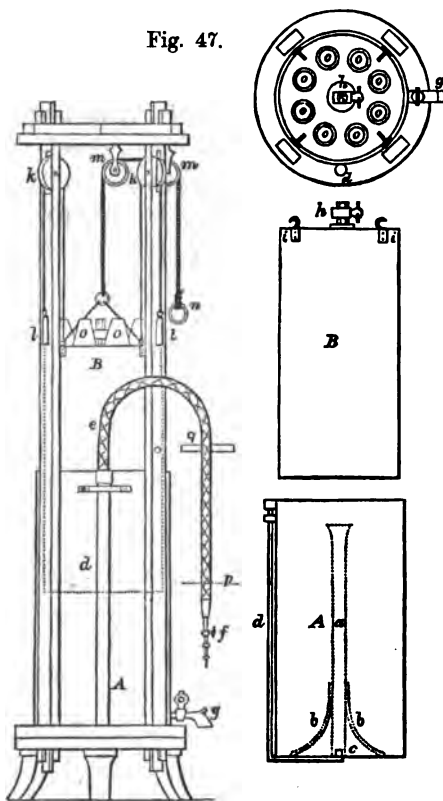
1. It should be perfectly airtight, under the greatest pressure used in Blowpipe experiments.
2. It should possess an orifice fine enough to afford an equal, uninterrupted, and sufficiently sharp stream of air.
3. That the pressure may be increased or diminished at pleasure.
4. That a long flexible tube be employed for conducting the stream of air, which is not in connexion with those parts of the apparatus which move up and down.
5. That it occupies as little room as possible.
6. And that, during the blowing, it is not required to fill it with air by means of a treadle, or other contrivance requiring physical exertion.

If such an apparatus be considered essentially necessary, the chief object of the application of the Blowpipe is lost ; on this account it should be premised, in the first instance, that this apparatus should be employed only by such as are well ac-

quainted with the use of the mouth Blowpipe ; and even then only as an auxiliary, or to preserve health in case inconvenience to the chest should arise from blowing ;—because, if a person, unaccustomed to the use of the ordinary Blowpipe should be sometimes obliged to employ it when travelling, his results would always be uncertain.

A Blowing apparatus, represented in Fig. 47,—constructed according to a model of a gasometer employed by LAMPADIUS, and which, with some slight changes, is similar to that proposed by HARKORT, possesses all the above qualifications.

Fig. 47.



It consists of two cylindrical vessels of strong sheet zinc, A and B ; A is two feet two inches high, and one foot two inches in diameter ; the inner B is two feet one inch high, and one foot one inch in diameter. Instead of zinc, copper, or tinned iron, may be employed. The last, however, does not last nearly so long as zinc, as it is liable to oxidize unless well protected by paint or varnish.

Through the middle of A, a brass tube *a*, with a funnel-shaped mouth, rises perpendicularly to the height of one foot

eleven inches ; it is secured in its place by two slips of strong tin plate *b b*, soldered to its sides and to the bottom of the vessel. To

the end of this tube another, of a less diameter, *c*, is attached, which passes externally underneath the bottom and up the side of the cylinder *A*, and is fastened at the top with a slip of zinc. To the top of the tube *c*, a strong caoutchouc tube *e*, three feet long and half an inch in diameter, is fastened, having at its extremity a brass stopcock *f*; it is to this that the various Blowpipe nozzles are affixed. A brass stopcock *g*, is also placed near the bottom of the vessel *A*, for the purpose of drawing off the water.

To the bottom of *B*, which should be strengthened internally with a double cross of tinned iron, is attached a brass stopcock *h*, having an orifice of half an inch in diameter, for admitting air. Four hooks are also attached to the bottom, to which the counterpoises for steadying the apparatus are fastened.

Both vessels are placed in a strong frame of hard wood, six feet high; the smaller, *B*, being inverted in the greater, *A*, which should be filled with water to about sixteen inches. This frame consists of a wooden support, six inches high, and one foot seven inches in diameter, into which four uprights, two inches broad and one inch thick, are mortised, lying with their broad sides close to the vessel *A*, and joined above to a framework similar to the base. In the base of the frame is a hollow, into which the part of the tube *c*, passing under the bottom, fits, so that the cylinder *A* may rest steadily on its support.

A wooden pulley *k*, is placed in each of the four uprights, just under the top cross pieces, over which cords pass attached by the hooks to the bottom of the vessel *B*, and having on the other ends leaden counterpoises *l*, each a pound weight. The counterpoises serve to prevent the vessel *B* from turning round on its axis, and thus keep the stopcock *h* always in the same direction, so that it may be conveniently opened and closed.

Two iron pulleys, *m m*, are also attached to the upper cross pieces, over which a strong cord, fastened to the centre of the bottom of the cylinder *B*, passes, having on its other end a strong brass ring *n*, with which the vessel *B* is drawn up.

If the vessel *B* be completely immersed in the water of the cylinder *A*, it may be filled with air by opening the cock *h*, and shutting the cock *f* on the caoutchouc tube, then drawing the

vessel up by the cord and ring above mentioned, until it nearly reaches the surface of the water, and shutting the cock *h* through which the air entered.

The air enclosed in this manner, which measures 3317·5 cubic inches, can be compressed by means of eight leaden weights, *o*, each of four pounds weight, in the form of a frustrum of a cone, so as to produce, through a Blowpipe nozzle placed on the stopcock *f*, an equal and sufficiently strong stream of air for all Blowpipe experiments. The nozzle must, however, have a larger diameter than those employed with the ordinary Blowpipe. The proper diameter cannot be well described here ; but the operator soon discovers the most convenient to use.

If the vessel *A* be filled with water to sixteen inches, the vessel *B* raised nearly to the surface, and the weights laid on, of the 3317·5 cubic inches of air only 2123·3 cubic inches escape through the stopcock *f* in a compressed state ; the remaining 1194 cubic inches remain behind in their usual state, as the level of the water cannot be raised higher than sixteen inches.

When, in experimenting with the instrument, a nozzle which answered best for the examination of silver and copper assays, was employed, the 2123·3 cubic inches of air were blown out in fifty-two minutes ; when a nozzle with a larger orifice, which was best suited for lead and tin assays, was used, it occupied thirty-five minutes before the vessel *B* had completely sunk down.

As the blowing is never continued so long in any analysis, the stopcock *f* should be closed when the blast is not required. In this way, it will not be often necessary to fill the apparatus more than once in an hour or an hour and a half.

The apparatus, as now described, may be placed in the laboratory at the right side of the Blowpipe table. The flexible tube, when not employed in blowing during the analysis, may be laid aside at the near right hand corner of the table ; when it is required, the stopcock *f* may be conveniently opened, and the tube held at *p*, between three fingers, like a pen, and the orifice placed in the flame of the lamp. When the apparatus is not in use, the flexible tube may be suspended to the lifting ring *n*.

This apparatus would also serve to form such small glass tubes

as may be useful in the examination of minerals, *et cetera*, before the Blowpipe, by using a lamp with a thick wick. The compression of the air being also diminished by the removal of some of the weights, it would be also necessary to employ a nozzle with a larger orifice than that used in analytical operations. In this case, both hands may be left disengaged, by fixing the stopcock *f*, on a small support, in such a manner that the current of air can be directed on the flame in a proper manner.

In conclusion, it will be best to state in what manner the vessel *A* is filled with water. The vessel *B*, being deprived of its weights, is drawn up as far as the frame admits, or till the stopcock *h* comes in contact with the central iron pulley *m*; it is kept in this position by two wooden pins, which are inserted into two corresponding holes in the front uprights; the funnel-shaped orifice of the upright tube *A* is then covered with a glass, and water poured into *A* until it stands at sixteen inches; it should not be filled higher than this, as the pressure of the air in the upper vessel, *B*, would cause the excess to flow over.

When the vessel is full, the glass is removed, the wooden pins withdrawn, the vessel *B* allowed to descend, and the weight placed upon it. If the vessel is made of well japanned sheet iron, cold water may be used to fill it; but if of zinc, copper, or unglazed iron, hot water should be employed; as otherwise, in a warm room, aqueous vapor would be condensed on its external surface, producing oxidation. Although this oxidation in itself would matter nothing, the vessels would lose much in external appearance,—the zinc becoming white, and the copper green. By adding one-fourth of boiling water, this unseemly appearance may be obviated.

QUANTITATIVE ANALYSIS

WITH THE BLOWPIPE.



*Preparation of the Substance, the Metallic Constituents of which
are to be ascertained.*

BEFORE it is possible to proceed with the investigation of a mineral, ore, furnace product, *et cetera*, in order to ascertain the different metals present, they must undergo a certain treatment, which is termed "Preparation of the Assay."

The antecedent labors are—desiccation of those substances containing mechanically mixed water ; and pulverization, when required in a finely divided state. Brittle substances, difficultly reducible to powder, may be crushed under the hammer upon an anvil : but malleable bodies can be first laminated, and then cut into shreds with a strong scissors.

It frequently occurs that ores, prepared on a larger scale, are apparently dry, although some per cents of water may be mechanically held between their layers ; in other cases, ores kept, after desiccation, in damp apartments, or in open vessels, reabsorb moisture from the air. If, therefore, the substance under examination contains mechanical moisture, a quantity greater than is requisite for two assays should be heated in a porcelain basin over a spirit-lamp, to expel the water, and the desiccated mass then triturated. During the desiccation, care must be taken not to raise the temperature so high as to roast the specimen.

Minerals and furnace products received for investigation in a dry, but not pulverized state, may be broken into fragments upon the anvil, and afterwards, if they be fit for powdering, triturated

in the agate mortar. Great exactness in the results cannot be expected, unless a portion eight or ten times greater than is required for one experiment be prepared,—except in the case of pure crystals and homogeneous specimens of minerals. It is impossible to represent the average constituents of any substance, as a rich metallic ore associated with extraneous nonmetallic constituents : for by taking a small portion for investigation, it is probable that either too small or too large a per centage of the metals is obtained, according to the excess of the metallic or nonmetallic ingredients present. Therefore, of minerals dressed on a large scale, an ounce should be selected from various parts of the mass, pulverized in an iron mortar, and, if possible, reduced to powder. A satisfactory result will be obtained, if 8 or 10 decigrammes, the quantity necessary for an experiment, be employed.

DESCRIPTION OF THE VARIOUS QUANTITATIVE ANALYSIS.

All minerals, ores, and furnace products must be specified under various heads, *videlicet* :—

¶ A. Ores, Minerals, and Products of Smelting Works,—the subdivisions of which are :—

- a. Such as contain volatile ingredients ;
- b. Such as contain no volatile ingredients except chlorine ;
- c. Compounds of metallic oxides reducible upon charcoal ;
and
- d. Such as are irreducible with borax and *workable lead*.

¶ B. Metallic Compounds, the principal ingredients of which are :—

- a. Silver,
- b. Gold,
- c. Copper or Nickel,
- d. Lead,
- e. Antimony or Zinc,

- f. Tin,
- g. Mercury,
- h. Iron or steel.

I.—THE SILVER ASSAY.

The Silver Assay with the Blowpipe, which HARKORT has published, is one of the most important quantitative analysis that can be performed with this instrument. It affords the means not only of ascertaining the proportion of silver in any ore, mineral, or production of smelting works, *et cetera*, but also of determining its quantity with sufficient accuracy. However, to obtain satisfactory results, it must be considered what ingredients besides silver are combined in the body under examination.

¶ A. *Ores, Minerals, and Furnace Products.*

a. EXAMINATION FOR SILVER IN THOSE MINERALS CONTAINING VOLATILE INGREDIENTS.

To this class belong, besides the ores prepared on a greater scale, and containing large quantities of Sulphides of Iron, Copper, Arsenic, Antimony, and Zinc, the following minerals: Vitreous Silver,—Sulphide of Silver,—Antimonial Silver, Melan Glance, light and dark Ruby Silver, Telluric Silver, Arsenical Silver, Argentiferous Sulphide of Copper, Miargyrite, Fahl Glance, Bismuthic Lead Ore, Vitreous Copper, Bournonite, Tin Pyrites, Galena, *et cetera*; and of the furnace products, *Rohstein*, *Bleistein*, *Kupferstein*, *Kupferleg*, *Tutty*, Lead Speiss, Cobalt Speiss, *et cetera*.

After preparation in the manner given on the two previous pages, the following procedure is to be undertaken :—

Weighing and Dressing of the Assay.

The weight is to be ascertained either on a balance, or upon the scales, described after the Gold Assay, and at the end of this Section.

Of rich silver ores associated with nonmetallic constituents, and therefore prone to represent very varying contents, two, or if necessary, three samples, of one decigramme, ought to be weighed twice; but poor silver ores, crystallized minerals, and also products of smelting works, which generally form a homogeneous mass, may be weighed once only. However, if the experimenter be not accustomed to Blowpipe examinations, even ores which generally do not materially differ should be weighed and examined twice.

A sample of one decigramme being weighed, it is to be poured from the basin of the balance, the adhering particles can be removed with the hair brush, so as to incur no loss,—into the mixing capsule, and dressed with borax and proof lead. The requisite proportion of borax varies according to the quantity and fusibility of the substance. In most cases, one decigramme is sufficient even for a difficultly fusible alloy; if, however, during the operation, the mass with this quantity appears intact when submitted to a strong heat, another small portion of borax should be added. For ores easily fusible, particularly for such as are not associated with earths, and consist of sulphides which unite quickly with lead, but do not oxidize so readily as this metal, a small quantity only of borax, 50 to 75 milligrammes, ought to be taken. However, if argillaceous constituents be in excess, or the assay contains much iron, cobalt, or tin, one decigramme of the flux will be necessary.

With regard to the lead, it should first be considered what other metals besides silver are extant in the alloy, for if an ore, mineral, or furnace product contains no more than 7 per cent. of copper, or 10 per cent. of nickel, five decigrammes of lead will be sufficient for one decigramme of the ground ore; but, on the contrary, if the substance contains more than the above cited per

centage of copper or nickel, the quantity of lead must be increased. As it is impossible always to have prescience of the per centage of these metals in a mineral or ore, the safest plan to adopt is to have a slight excess of lead, otherwise the copper cannot be perfectly detached from the silver, and also, a fusion of a nickeliferous *workable lead* is nearly impossible.

The annexed table shows the proportions of lead to the substance in some of the most common minerals and furnace products, consisting partly of copper and nickel.

	NAMES OF THE MINERALS.	PER CENTS. OF METAL.	DECIGRAMMES OF LEAD.
ONE DECIGRAMME OF	Copper Glance.	84 Copper.	15 Decigrammes.
	Purple Copper	63 "	12 "
	Tennantite	45 "	10 "
	Copper Blende	42 "	10 "
	Grey Copper	40 "	10 "
	Cupreous Bismuth	35 "	10 "
	Copper Pyrites	34 "	10 "
	Argentiferous Copper Glance .	30 "	10 "
	Tin Pyrites	28 "	7 "
	Bournonite	12 "	7 "
	Kupferstein, obtained from the fusion of roasted Cupri- ferous Bleistein	45 "	10 "
	Kupferleg	50 "	10 "
	Lead-speiss	50 Nickel, Cobalt, and Copper . .	10 "
	Cobalt-speiss	55 Nickel and Cobalt.	10 "

Having added the necessary proportion of borax and lead to the ground ore, the whole assay is to be mixed, the handle of the spoon serving as a spatula. A cornet of soda paper is now held carefully between the thumb and fore-finger of one hand, the capsule with the assay in the other; its contents are carefully placed in the cornet, and the adhering particles detached by the small brush, and added to the main mixture in the recipients. When this is accomplished, the sides of the upper part of the cornet must be brought together, folded over about a quarter of an inch, and pressed; care must, however, be used in closing it, lest the under part be broken, and particles of ore be lost. This accident will be avoided by keeping the cornet upon the long side of the middle-finger, during the filling process.

The cornet, filled with the assay, is next placed upon a porcelain basin, where it is to remain until required for the experiment,—and if the same substance is to be investigated several times, or various ores are to be examined for silver, they must be weighed and dressed similarly. The operator should remember, that the weighing of the assay must be followed immediately by the dressing with borax and lead, and should different substances be examined, a superscription on the cornets will prevent any mistake. The weighing and dressing is succeeded by,—

The Fusion of the Assay.

This is effected upon charcoal, in the reducing flame. First, a cylindrical cavity, the size of the filled cornet, is bored with the instrument described in the first Section,—page 25, Fig. 24,—into a good piece of charcoal, upon its diametrical section, close to one of the edges. In this deepening the cornet is placed, and carefully adjusted, so that it touches on all parts.

A strong reducing flame is now to be directed upon the whole surface nearly of the assay. Although the soda paper becomes carbonized, still this carbon is not destroyed, until the upper part of the borax, together with the particles of ore, are fused; and when this is accomplished, it is impossible to remove them by the current of air. If, after the combustion of the carbon, portions of

the assay appear as liquid scorïæ, mingled with lead globules, the whole must be enveloped in a strong and pure reducing flame. During this operation, a small portion of sulphur, arsenic, antimony, zinc, *et cetera*, volatilizes, but the greater portion fuses, and coalesces into a bead with the lead, *et cetera* ; while the argillaceous ingredients, with small quantities of the nonvolatile metals, oxidized, unite with the borax, forming scorïæ. Sometimes, when the alloy consists of difficultly fusible metals, the scorïæ appear to be perfectly free from lead, although, in its under part, the substance remains intact ; to submit this to the action of the flame, the charcoal must be inclined to the other side, and thus a further flowing action takes place in the cavity, and the sample is reversed.

By this turning, which cannot be spared even with the most easily fusible mixture, the bottom of the soda paper is brought to the upper part of the charcoal, and, as it will be difficultly consumed by a pure reducing flame, the assay should be placed in such a position to the flame, that the recrements are only covered by it where they are not in contact with the soda paper ; by observing these directions, atmospheric air accedes, and the cornet is speedily consumed. When this occurs, the whole sample must be again covered by the flame, in order to reduce and reunite with the main bead all traces of lead, which during the antecedent process might have oxidized and combined with the recrementitious matter.

If the scorïæ which have been enveloped in the reducing flame appear orbicular, perfectly fluid and free from lead, although their position near the bead of lead had changed several times, the operator may infer that they are free from silver also.

During the operation, it is not indispensable to keep the leaden bead wholly covered while the scorïæ are subjected to the reducing flame, but the temperature must always be sufficiently high to keep the plumbiferous globule fluid. However, if, owing to an imperfect reducing flame, it happens that the recrementitious particles spread over the charcoal, presenting small globules of lead, the main bead must be covered wholly by the flame, and, by inclining the charcoal, brought to those parts of the support

where the small ones are visible, in order to combine with them. The assay being treated, as before mentioned, so far that the scorizæ are in full fusion, and free from lead, the reducing must be substituted for an oxidating flame, which must be kept at a somewhat greater distance from the lead globule. By the judicious application of the flame, the volatile metals, together with the sulphur, separate from the lead, and the readily oxidizable ones, as iron, tin, cobalt, likewise a small portion of nickel and copper, combine with the scorizæ, partly as protoxides, and partly as sesquioxides, whereas the greater portions of the nickel, copper, and silver remain with the lead.

After elimination of nearly all the volatile constituents, the lead and also traces of silver rapidly oxidize;—the latter, even in rich ores, is almost imperceptible, and becomes still more insignificant, when the scorizæ containing the oxide of silver touch the hot charcoal, for the greater portion of it is again reduced, and can be made to recombine, by the movement of the scorizæ with the argentiferous lead globule.

So soon as all volatile ingredients are vaporized, the motion and oxidation of the lead increase rapidly, attended with considerable ebullition of the scorizæ. By inclining the support, the button, which is generally surrounded with recrement, is brought to a free place, and allowed to refrigerate. After cooling, if the argentiferous lead,—*workable lead*,—obtained be of a white color, the operation is completed; but if it appears dull or blackish, traces of sulphur are present, and these must be expelled by repeated oxidation. Great care must be taken, in the expulsion of the volatile ingredients; for, in the first place, the brittleness of an impure workable lead might prevent it from being removed from the scorizæ, without loss of some of the particles; and, secondly, not only because it can with difficulty be cupelled, but particularly, if sulphur remains, a violent motion ensues in the operation, occasioning the projection of some of the metal from the cupel.

If the regulations here laid down be strictly followed, the small lead globules remaining in the scorizæ will not be perceptibly argentiferous, as they result only from the oxidation of the main

bead, and reduction of the oxide from the silverless recrements by the carbon. Although the small quantities of silver oxidizing in company with the lead are to be calculated as loss, still they, in comparison to the oxidized quantity of lead, do not surpass the proportion of silver lost in the beginning of the oxidation with the same quantity of lead ; as will be shown after describing this operation.

Of substances treated in this manner, the most difficult of fusion are, sulphides of iron, arsenic, some ores of nickel, cobalt, and a species of *rohstein*, principally consisting of sulphide of iron ; whereas other substances appertaining to the same class generally melt readily, even when they contain difficultly fusible earths.

The fusion of these substances is greatly facilitated by roasting on charcoal, a process to which the lead assays are generally submitted, with subsequent addition of the borax and lead necessary. In this operation, the greater part of the arsenic and sulphur volatilizes, and the remainder becomes acidified, combining with the newly formed oxides of cobalt and nickel. By submitting this assay to the inner flame, the oxide of nickel is reduced, the sesquioxide of iron becomes protoxide, and the acids are reduced to sulphur and arsenic. The metallic nickel combines with the lead, together with the silver and traces of sulphur and arsenic, forming an easily fusible alloy ; the free portions of sulphur and arsenic volatilize, and the sesquioxide of iron and oxide of cobalt dissolve in the borax.

When the scorixæ appear perfectly molten, and free from lead, it is only necessary to submit the alloy for some moments to the outer flame, when the last traces of sulphur and arsenic will be expelled.

The fusion of argentiferous minerals should always be accomplished in the reducing flame, because if the assay be treated with the oxidating flame, exact results cannot be expected : a considerable portion of the lead would oxidize at the outset, dissolve in the borax, and, coming in contact with the carbon, be reduced by it, forming globules with small particles of silver, which are intermingled with the recrements ; and even if these reunited with

the main bead, they would soon be replaced by others newly formed, undistinguishable from the argentiferous lead.

The time necessary for fusing a non-previously roasted alloy varies from five to eight minutes, according to its fusibility and the quantity of volatile and argillaceous constituents.

The refrigerated globule must be separated from the scorise in the following manner :—The whole mass should be removed with the forceps, wrapped in paper, and placed upon the steel anvil ; by a few gentle strokes of the hammer, the recrementitious particles are perfectly detached.

Treatment of the Workable Lead obtained by the Fusion.

The separation of lead from silver contained in *workable lead* is effected by oxidation at a red heat, with access of atmospheric air, and is based upon the property of lead to oxidize at such a temperature, while the silver remains unaltered. HARKORT divides this process into two stages, the first termed *Oxidation*, the second, *Cupellation*.

The Oxidation.

This operation is a very simple process. . A cupel of *sieved* bone ashes is prepared by means of the instrument described at page 29, placed upon the mould, and submitted to a red heat in the oxidating flame, to remove all hygroscopic moisture. By this precaution the experimenter will avoid a loss of workable lead.

The cupel being desiccated, is charged with the workable lead, and smelted in a strong outer flame until the surface of the assay brightens, and oxidation commences. If the *workable lead* contains large portions of copper or nickel, the fusion requires more time, owing to the nickel separating from the lead at the commencement, and covering its exterior with an infusible layer, which prevents access of atmospheric air, and makes the oxidation impossible,—the copper renders the alloy but difficultly fusible. If, therefore, much nickel is present, a small quantity of pure lead, —two to four decigrammes,—should be added to the assay.

When the oxidation begins, the point of the Blowpipe must be advanced so as to produce a fine blue flame, which must be directed upon the sides of the cupel, in order to continue the fusion without immediate contact ; by this procedure the surrounding air has access to the bead, the lead and copper of which oxidize, forming scoriæ, which are conveyed on the liquid mass to the sides. If the assay does not contain much silver, the scoria iridesces beautifully, and remains upon the sides of the cupel, forming a solid mass—*litharge* ; on the contrary, if the alloy be rich in silver, no prismatic colors appear, so that from this phenomenon a conclusion is arrived at, with regard to the richness or the poor-ness of the ore. The presence of copper renders the litharge almost black ; but if the assay be free from this metal, the dross is of a reddish-yellow color.

Care must be taken during this process to preserve an appropriate temperature, for if the heat be too high, portions of lead volatilize, particularly in rich assays, entrailing a small portion of silver, and the litharge, instead of congealing, remains fluid, and is absorbed by the bone ashes of the cupel, thus causing a new loss of silver. On the other hand, if the temperature be not sufficient for keeping up the oxidation, a coating of litharge covers the exterior of the alloy, and checks oxidation, *et cetera*. The loss of silver caused by this oversight is nearly imperceptible, as the oxidation again proceeds when the temperature augments ; but this accident should nevertheless be avoided.

If the oxidation be effected at the temperature at which the litharge surrounding the bead solidifies, and accumulates in such a manner that it covers the greater part of the assay, obstructing the free access of air, then the cupel must be inclined, that the molten lead may flow by its own gravity to the sides,—thus acquiring a greater oxidating surface. Its volume now diminishes rapidly, and when it is in a poor assay, of about double the size represented in Fig. 6, c, page 11, or in a rich ore, the operator must carefully regulate the distance between the cupel and the flame, for the gradual cooling of the assay. The slow refrigeration is necessary to obtain the globule in a regular form, and prevent any loss of silver, for by too sudden a decrease of tempera-

ture, the lead generally detaches itself at once from the scorix, often occasioning the projection of particles of silver from the cupel.

A phenomenon peculiar to rich alloys, which ensues if a sample containing upwards of fifty per cent. of silver be treated so far that the proportion of silver to lead is about seven to one, ought not to be omitted : in such a case, the congealing globule acquires a greyish-white coating, containing a large proportion of silver ; probably this is a suboxide of lead, with metallic silver.—The greater portion of this pellicle remains with the litharge, causing a considerable loss of the noble metal. If this inconvenience occurs, the assay ought to be fused again with the reducing, and afterwards submitted to the oxidating, flame, in order to change the above given proportions. When, therefore, rich substances are to be examined, the process must be pursued until the globule consists of pure silver nearly, as the previously mentioned phenomenon only manifests itself in alloys of the composition one part lead to seven of silver.

The operation being regularly terminated, the cupel is removed from its support, put upon the anvil, and the litharge with the argentiferous globule, placed in a porcelain basin. During the refrigeration, the operator can prepare the cupel for the succeeding process.

The separation of the metallic globules from the dross is most easily accomplished, by placing the mass upon the anvil in a strong paper, and striking it rather sharply with the hammer. In this process also, the globule acquires an indentation, which admits of its being placed in the cupel, so that it cannot move from place to place.

The Cupellation.

The cupellation is the most difficult part of the whole experiment, and, for obtaining exact results, requires great care, and also long practice. One of the principal requisites is a good cupel, possessing a smooth surface, no fissures in the interior, and not too dense, as it is necessary, in this operation, that the bone ashes

should absorb all the litharge generated. For this reason, bone ashes not only sifted, but also levigated, ought to be employed,—the first not affording so dense a mass, whereas the latter, by themselves, are apt to give a soft surface.

The preparation of the cupel is conducted in the following manner :—The oxidation process being finished, and the argentiferous lead, *et cetera*, removed from the bone ashes, the cupel not spoiled by litharge is to be comminuted with the iron spatula, and then placed in the iron stand, the cavity filled with levigated bone ashes, and the new cupel formed by means of the bolt ; the cupel is then submitted to a red heat. If any crevices, owing to moisture, open during the process, they are readily filled up by applying, with some force, the warm clean bolt, upon the interior.

When the cupel is finished, the argentiferous globule must be placed close to its border, and the support then approached near the flame. An oxidizing flame having been directed horizontally upon the button until fusion has ensued ; the support must then be brought gradually to a perpendicular position, by which motion the globule leaves its place and rolls to the centre of the cupel. At this moment the flame is withdrawn from the bead, and directed upon the cupel only, surrounding it, which must be kept up at a red heat during the experiment. If the temperature be sufficiently high, the bead will remain molten, without being touched by the flame ; but if not, the assay must be again fused with the open flame, and afterwards subjected to the other treatment.

The surest guarantee for the success of the cupellation is the dryness of the bone ashes, and the absorption of the litharge, for when the bone ashes are not sufficiently heated, the cupel acquires a coating of litharge, upon which the globule oscillates, and if the assay be not altogether spoiled, at least the results will be uncertain. For the completion of the operation, the heat is augmented for the purpose of expelling entirely the last coating of litharge ; the globule must then be withdrawn gradually from the flame, and allowed to cool. An inspection through the lens will then convince the operator whether the surface is bright, and if not, a new cupellation must be undertaken. In rich assays, five to

ten seconds before the brightening, iridescence ensues,—a beautiful play of colors appearing momentarily on the surface of the globule, which after a short time, appears lustrous and white,—which disappears with the remainder of the litharge. It is, however, necessary to continue the blast, and the touching the button on all parts with the apex of the blue flame, until the silver exhibits a pure bright color. In the heating of a greater globule, small asperities sometimes appear on the exterior, which might be taken for the extraneous matters, but after refrigeration they will be recognized as silver. When silver obtained from chloride of silver is treated with the oxidating flame, after the lapse of a few seconds, asperities present themselves, which, however, cannot be removed either by the reducing flame or upon charcoal. A partial oxidation of the silver appears to take place in this instance.

To prevent a *spitting* of the metal, the globule must, as previously remarked, be cooled very gradually.

The cause of this spitting is, according to LUCAS and GAY-LUSSAC, owing to silver in the molten state being capable of absorbing large portions of oxygen gas from the air, which is rapidly discharged during the refrigeration of the metal, and sometimes occasions the projection of small portions of silver from the cupel. GRAHAM states that this property is possessed by pure silver only, and does not appear at all in silver containing one or two per cent. of copper.

Should the assay contain copper, the silver, at the moment of brightening, generally dilates upon the cupel, and although it appears white after cooling, yet this is no proof of its purity. Such a button ought to be fused and cupellated a second time, with one decigramme of pure lead, if it is large enough for weighing, and if not 50 milligrammes, in order to obtain a pure and splendid globule. The cupellation of a cupriferous alloy in this manner is of better effect, than by the direct addition, at the outset, of a quantity of lead sufficient for the separation of the copper, as this, in many cases, requires the double quantum of lead, impeding the oxidation, as well as the cupellation; the reason is, that in the oxidation much less copper oxidizes, comparatively, than in the cupellation.

In the cupellation of poor assays, inconveniences often arise, which appear to be of very little importance, but if attention be not paid to them, exact results will not be obtained.

1. It often happens that the small globule adheres to the cupel, and when the process is pursued, the form becomes so irregular that a determination of its weight is very difficult. When this occurs, a small portion of pure lead should be added to increase the weight of the assay, and the whole recupelled. If then the cupel be slightly inclined, the gravity of the button is sufficient to segregate it from the impediment, and conduct it to another place, where the process can be terminated.

2. Sometimes, if the operator does not possess sufficient practice in cupellation, the litharge, instead of being absorbed by the bone ashes, surrounds the metallic mass. Should this take place, the operation must be suspended, and the button, if large enough, removed by the forceps from the recrementitious matters; if, on the contrary, it be too small, a portion of pure lead is to be added, and fused with it. In both cases, the operation must be performed upon a newly prepared cupel.

3. Often, after brightening, a coating of litharge remains upon the silver, and, although the metal appears to be pure, cannot be entirely separated. Its expulsion and imbibition are, however, effected, by placing it at a convenient distance from the Blowpipe, and submitting it to a powerful oxidating flame. If all the directions given in the preceding pages are exactly complied with, it is possible to obtain accurate results, even from substances containing 0.000752 per cent. of silver.

In cases where it is necessary to find out the quantitative contents of alloys still poorer, several assays of one decigramme should be weighed with the requisite quantity of borax and lead, fused in the previously described manner, and cupellated like workable lead, two or three globules at a time. When this is finished, all the buttons must be united, and again submitted to a new oxidation and cupellation. By this process, a determination of the silver contained in the whole substance is effected, so that, by a simple division of the weight of the button by the number of decigrammes employed, the per centage of silver in each assay may be found.

Determination of the Weight of the Silver Globules obtained.

In order to determine the weight of the pure silver globule, it must be removed from the cupel by the forceps, and freed, as formerly shown, from any adhering litharge, then weighed upon the balance. Should it be so small that its gravity can be ascertained with greater exactness upon the scale, it must be very carefully removed from the cupel, so that its form may not alter, and measured on the instrument described after the Gold Assay.

As, in the cupellation of a larger quantity of argentiferous lead, a small portion of silver oxidizes, which, combining chemically with the litharge, is absorbed by the bone ashes, it was deemed necessary to ascertain this loss, to ensure an accurate result.

Although this oxidation of the silver takes place, not only in the cupellation, but also in the first process, the loss sustained is much inferior to that occurring in the mercantile assayings, in which all the litharge is imbibed by the cupel.

In an assay containing one per cent. of silver, it is almost impossible to determine this loss upon the balance; it, however, increases in a relative proportion with the size and nature of the globule. It also varies according to the quantity of lead employed; but remains constant for each per centage of silver, if the proportions of the lead, and the temperature, are not changed.

The following will show the justness of these observations, in contradiction to the statements of those chemists, who suppose that a mechanical separation of the silver is effected.

1. If a button of pure silver, of known weight, be fused in the oxidating flame, with a portion of litharge upon a clay basin, and, after refrigeration and separation from the scorix, reweighed, a loss of silver will be obvious, although no traces of metallic silver are perceptible in the litharge, or upon the basin. If the litharge is mixed with a small quantity of soda, and treated in the reducing flame, a plumbiferous globule will be obtained, which, when cupelled, affords a silver button. Gold, when submitted to the preceding treatment, suffers no loss, and, therefore, no trace of the noble metal is obtained on cupelling the litharge.

2. If a weighed silver globule be heated with borax upon charcoal, in the oxidating flame, the borax glass assumes an enamel aspect, after the lapse of some moments, owing to the solution of some oxide of silver. When this glass is heated in the reducing flame, small particles of metallic silver appear upon its exterior. Though this last example does not prove that the loss of silver sustained in the cupellation is chemical, it shows the capacity of this metal to oxidize at a high temperature.

These reasons were sufficient inducements to the formation of a Table for the use of the assayer, that he might readily detect the quantity of silver lost in any of the alloys. This Table is not only adapted for the determination of the loss sustained in the cupellation of substances free from copper, but also for cupriferous alloys, which are not obtained in a pure state by the first addition of lead. The proportions, with the requisite quantities of lead for cupellation are given; *videlicet*, instead of 5, 7, 10, 12, and 15 decigrammes—see pages 301, 302,—6, 8, 11, 13, and 16 should be taken—see page 315. The following suggests itself here: whether, in a cupriferous silver ore, requiring, say 15 decigrammes of proof lead, and treated, according to the quantity of sulphur, in the oxidating flame,—by which a part of the lead oxidizes, combining with the scorise, and another part volatilizes,—the loss in the cupellation is the same as if no lead was lost in the fusion? Experience affords the following answer:

If an argentiferous lead be treated with borax on charcoal in the oxidating flame, a small portion of silver oxidizes with the lead, and this loss of silver being in an exact ratio to the loss sustained by *oxidation*, where the litharge remains on the cupel, this loss of lead must therefore be taken into account.

The loss of lead by volatilization, which sometimes amounts to half or one decigramme, needs not, however, be considered, as the volatilized portion causes no appreciable change in the cupel loss, on account of the difference produced in such cases, even where the per centage of silver is very large, not exceeding 0.01, or, at the utmost, 0.05 milligrammes.

Compensation Table for the Loss of Silver sustained in Cupellation.

Weight in Milli- grammes of the silver globule obtained by cupel- lation.	If the assay contains, of copper,					If the assay has under 7 per cent. of copper, or does not contain this metal at all, it must be dressed with, <i>Decigrammes of lead,</i>				
	80 to 90 per Cent.	60 to 79 per Cent.	30 to 59 per Cent.	10 to 29 per Cent.	7 to 9 per Cent.					
	It is to be dressed and oxidized with, <i>Decigrammes of lead,</i>									
	16	13	11	8	6	5	4	3	2	1
The loss of Silver by absorption amounts, in <i>Milligrammes</i> , to										
99.75 to 99.5	0.50	0.45	0.39	0.32	0.25
90	0.88	0.69	0.47	0.42	0.36	0.29	0.22
80	0.75	0.64	0.44	0.39	0.33	0.26	0.20
70	0.82	0.68	0.58	0.40	0.35	0.29	0.23	0.18
60	0.74	0.61	0.52	0.36	0.30	0.26	0.20	0.16
50	0.65	0.54	0.46	0.32	0.26	0.23	0.17	0.14
40	0.62	0.55	0.46	0.39	0.27	0.22	0.20	0.15	0.12
35	0.57	0.50	0.42	0.36	0.25	0.20	0.18	0.13	0.11
30	0.51	0.45	0.38	0.32	0.22	0.18	0.16	0.12	0.10
25	0.45	0.40	0.34	0.29	0.20	0.16	0.14	0.10	&c.
20 . .	0.45	0.39	0.35	0.29	0.25	0.17	0.14	0.12	&c.	
15 . .	0.37	0.32	0.28	0.23	0.20	0.15	0.12	0.10		
12 . .	0.32	0.26	0.23	0.19	0.17	0.13	0.11	&c.		
10 . .	0.27	0.23	0.20	0.17	0.15	0.11	0.10			
9 . .	0.25	0.21	0.18	0.16	0.14	0.10	&c.			
8 . .	0.22	0.18	0.16	0.15	0.13	0.09				
7 . .	0.20	0.16	0.14	0.13	0.12	0.08				
6 . .	0.17	0.14	0.12	0.11	0.10	0.07				
5 . .	0.14	0.12	0.11	0.10	0.09	0.06				
4 . .	0.11	0.10	0.09	0.08	0.07	0.05				
3 . .	0.09	0.08	0.07	0.06	0.05	0.04				
2 . .	0.07	0.06	0.05	0.04	0.04	0.03				
1 . .	0.05	0.04	0.04	0.03	0.03	0.02				

Here it is scarcely necessary to observe, that if the operator be not accustomed to Blowpipe operations, the cupellation is generally effected at too high a temperature, in which case, even if account be kept in mind of the cupellation loss, too low a percentage of the noble metal ensues. It is, therefore, of the greatest importance to ascertain the proper degree of heat required for the experiment, and the best way to arrive at it is by repeated analysis. The following is given as an example :—

A globule of fine silver is exactly weighed, dressed with five decigrammes of lead and the requisite proportion of borax, fused in the reducing flame, and the workable lead obtained, cupelled. If no mechanical loss ensues, and by weighing the button a greater deficiency is indicated than given in the Table, the temperature is too high ; but if the loss corresponds with that noted in the Table, the necessary heat has been employed. The greatest loss is generally sustained in cupellation.

If the balance is so sensible that it indicates a weight of 0.05 milligramme, the *cupellation loss* may be carried to the second place of decimals, and calculated for a silver button the weight of which is *between* 90 and 100, 80 and 90, or 70 and 80, *et cetera*, milligrammes, from the difference of the same ; *exempli gratia*, if, from an assay dressed with 5 decigrammes of lead, a silver button is obtained having a weight of 53.45 milligrammes, as the difference between 50 and 60 is 10, and as 53.45 makes the third part of this difference, so the “cupel loss” for the percentage of silver would be $0.32 + \frac{0.36 - 0.32}{3} = 0.32 + 0.1 = 0.33$ milligramme.

If, however, the balance is only sufficiently delicate to turn with 0.1, the cupel loss should not be extended to the second place of decimals,—when the number in the second place surmounts 5, it is to be reckoned as 0.1.

It is obvious that the cupellation loss is not to be reckoned in experiments serving as a control in mercantile examinations, because a larger deficiency of silver results in such operations ; nor is it necessary with globules the weights of which are ascertained upon the scale, the loss sustained in them being so minute that its results may be exceeded by mistakes committed in measuring their diameters.

b. EXAMINATION OF ORES, MINERALS, AND FURNACE PRODUCTS CONTAINING NO VOLATILE INGREDIENTS EXCEPT CHLORINE.

To this class belong ores consisting principally of earthy matters, and poor in silver,—roasted argentiferous ores,—all silver ores roasted with chloride of sodium, which are to be amalgamated, and the residues of the amalgamated process; Horn Silver, Earthy Silver Glance, argentiferous slags, and also the dross of gold- and silversmiths.

Of the desiccated and pulverized substance, two-thirds of a decigramme are weighed, and dressed with one decigramme of borax and five decigrammes of proof lead. If, however, the assay contains copper, the proportion of lead must be augmented in proportion to the quantity of the former metal. The whole is then enveloped in a cornet of soda paper, and pressed into a prepared charcoal cavity.

As in this operation few constituents are present which are to be combined with and then separated from the lead, and as the process consists principally in forming an argentiferous button, and vitrifying the earths and difficultly reducible metallic oxides by means of the borax, the assaying is effected without the slightest inconvenience. The sample is only to be submitted to the reducing flame until the lead has completely alloyed with the silver, forming a molten button, and the recrementitious matters are in full fusion. The same precautions are to be observed as in the fusion of ores containing volatile ingredients. By this procedure, the earths and difficultly reducible metallic oxides are vitrified, the easily reducible ones, as the oxide of lead, of roasted lead ores, *et cetera*, reduced and combined with the proof lead, the chloride of silver decomposed, and the chloride of lead formed volatilized as a white vapor, part of which coats the charcoal; the silver fuses, and alloys with the lead, forming workable lead.

When the operation is terminated, the workable lead is allowed to refrigerate, then detached from the scorix, and beaten into a cube upon the anvil. The oxidation, cupellation, and the weighing or measuring, are performed in the ordinary way.

Should the substance contain more volatile ingredients than chlorine, the workable lead will appear of a black or dark grey color; when this occurs, it ought to be refused with the scoræ for some minutes in the oxidating flame, in order to expel any sulphur or arsenic that may be present. Ores containing very small portions only of real silver present no black aspect, as the minute qualities of sulphur and arsenic are evolved on the first application of the heat.

c. EXAMINATION OF FURNACE PRODUCTS CONSISTING OF METALLIC OXIDES WHICH ARE EASILY REDUCIBLE UPON CHARCOAL.

The most prominent of these are the *litharges* and the *abstrichs*.—If the workable lead resulting from plumbiferous ores be cupellated for silver, several sorts of litharge are obtained. The first appearing immediately after the fusion of the substance, is the *absung*, which adheres to plumbiferous minerals. The second are termed abstrichs, or *black litharges*, and present themselves when the molten mass is exposed to the blast; their aspect changes from black and metallic, to yellow and yellowish-grey, and they contain all the easily oxidizable metals; the metal generally predominating in them is antimony. The *red litharges* formed after the *abstrichs*, until the termination of the process, consist of nearly pure oxide of lead, contaminated only by traces of silver and oxide of copper.

Their content of silver is generally inconsiderable, and often cannot be ascertained in an assay of one decigramme. As, however, these substances consist of no foreign metal besides lead, the reduction of which is easily effected, their quantity of silver is readily determined.

Five decigrammes of the body are weighed, pulverized, and dressed with 50 milligrammes of soda, and 50 milligrammes of borax, enveloped in a cornet of soda paper, placed in a charcoal cavity, treated with the reducing flame, until all the oxide is reduced, and the scoræ, free from lead, are perfectly liquid.

The globule obtained from the litharge is generally exempt

from volatile metals, and contains but a small proportion of copper; whereas, that obtained from the abstrich is usually contaminated with antimony, arsenic, copper, zinc, *et cetera*. These ingredients, however, disappear, if the scoræ are treated for a long time with the reducing flame. The oxidation and cupellation are effected in the ordinary way. The weight of the button is determined by the scale.

d. EXAMINATION OF MINERALS WHICH ARE NOT DECOMPOSED BY BORAX OR LEAD UPON CHARCOAL.

Up to this time no mineral can be arranged under this head, except Molybdena Glance. It contains 0.176 per cent. of silver.

For analyzing this mineral, the following flux has been found the most satisfactory:—

Take 1 decigramme of the specimen, and dress it with—

Soda, 150 milligrammes;

Borax, 150 milligrammes;

Lead, 5 decigrammes.

In a cornet of soda paper inserted in a cavity in the charcoal, place the assay, and subject it to a strong reducing flame. The soda decomposes the mineral, sulphide of sodium is formed, and the liberated molybdenum partly combines with the lead, and partly volatilizes, coating the charcoal with a white sublimate. When the liquid scoræ flows quietly, it is a sign that the decomposition is terminated. The argentiferous globule must now be exposed to the air, and treated with the reducing flame, until all the molybdenum, which gives a white and brittle alloy with the lead, is expelled. The addition of the borax is to prevent the recrements from dilating upon the support. The workable lead obtained is then oxidated and cupellated.

¶ B. *Metallic Compounds.*

a. EXAMINATION OF THOSE COMPOUNDS WHICH HAVE SILVER AS A PRINCIPAL CONSTITUENT.

Among these rank native silver, *blicksilber*, *brandsilber*, *amalgamsilber*, cupelled silver, and standard silver. The operation for these consists only in fusing with pure lead, and cupellating. The quantity of the substance taken may vary from 80 to 100 milligrammes, as it is very difficult to obtain a piece of a certain weight.—As the examination is not effected upon exactly 100 milligrammes, a particular calculation is necessary for deducing the per centage of the assay from the weight of the treated substance; thus, 85.5 milligrammes were treated with 200 milligrammes of lead, from which 83.6 milligrammes of pure silver were obtained, the cupellating loss of which is 0.27 milligrammes, therefore, the following proportion is indicated :—

$$85.5 : 83.6 + 0.27 :: 100 : x.$$

$$x = \frac{(83.6 + 0.27) 100}{85.5} = 98.09 \text{ per cent.}$$

On account of such compounds being obtained with great difficulty in small particles, as foreign matters on the surface of the sample might prevent exactness in the results, they should be scraped off previously to weighing, and treated on charcoal. If native silver, *blicksilber*, or *brandsilber* is to be assayed, one decigramme of lead and 50 milligrammes of borax are requisite, for cupriferous amalgams and standard silver; the proportion of lead varies from two to five decigrammes, however, according to the quantity of copper. The mixture is fused in the reducing flame, until the noble metal becomes alloyed with the lead, and the borax appears perfectly free from plumbiferous particles. The assay is then allowed to refrigerate, and the button removed and separated from the flux. Though the addition of borax is not absolutely required, still it prevents any violent action ensuing, which, if occurring, would falsify the results.

Such alloys as require but one to two decigrammes of proof lead,

are assayed upon the cupel. Cupriferous alloys, however, treated with three to five decigrammes of lead, must first be submitted to oxidation, to dispel the greater part of the copper, and then cupelled.

The fine silver globule is, after the termination of the process, removed from the cupel, struck slightly with the hammer upon the anvil, and then weighed upon the balance.

b. EXAMINATION FOR SILVER IN METALLIC COMPOUNDS
CONSISTING CHIEFLY OF GOLD.

Under this head is placed Native Gold, and alloys consisting of gold, silver, and copper.

In these the per centage of silver is readily determined at the same time with that of gold. The procedure will be specified under the Gold Assay.

c. EXAMINATION FOR SILVER IN ALLOYS WHEREIN COPPER
AND NICKEL PREDOMINATE.

These compounds are :—*black copper* and *gaarkupfer* affined on a large scale ; copper coins containing silver ; brass, bell-metal, German silver, *et cetera*.

For procuring quantities sufficient for weighing, the alloy must be ground with a strong file, and when *gaarkupfer*, *black copper*, coined copper, or German silver is assayed, the following proportions must be employed :—

Substance,	1 decigramme ;
Lead,	15 decigrammes ;
Borax,	1 decigramme.

The dressed sample is then wrapped in a soda paper cornet, inserted in a charcoal cavity, and treated with the reducing flame directed upon the assay, until it has been some time in fusion, and all metallic particles have disappeared from the surface. In this operation, cobalt, iron, and zinc oxidize ; the first two dissolve in the borax, whereas the zinc volatilizes.

The workable lead obtained, which contains copper or nickel, or both metals, may, without being cooled, be brought upon the steel anvil. This spares the detaching of the scoriæ. It is now to be cupelled in the common way, but if the globule produced contains copper, a dilation ensues, which will prevent a determination of its weight upon the scale ; this is, however, remedied by adding 50 milligrammes of lead, and recupelling.

When brass or bell metal is to be assayed, the following proportions must be taken :

Alloy, 1 decigramme ;
Lead, 10 decigrammes ;
Borax, 1 decigramme.

The assay is fused like the former, in the reducing flame, until the borax is exempt from metallic particles. The flame is then directed only upon the flux, to allow the atmospheric air access to the lead, to oxidate the tin, and the nonvolatilized portions of zinc. The oxide of tin combines with the borax ; the oxide of zinc is entirely dispelled. If, then, the surface of the assay appears bright, it must be poured upon the anvil. The subsequent operations are conducted according to the method prescribed for cupriferous substances.

d. EXAMINATION OF METALLIC COMPOUNDS, THE PREDOMINATING METALS BEING LEAD OR BISMUTH.

To this class appertain workable lead, prepared on a large scale, revived lead, argentiferous bismuth, *et cetera*.

Of these alloys, the workable lead is the richest in silver ; a portion is to be laminated, cut into shreds with the scissors, two decigrammes weighed out, placed upon a cupel, fused in a moderate oxidating flame, and cupelled immediately. If little or no copper is present, a bright and orbicular globule will be obtained, but when much copper is contained in the alloy, dilation ensues, without any external brightening ; should this occur, one decigramme of lead ought to be added, and the operation terminated upon another part of the cupel. The weight of the button

obtained, divided by two, gives the per centage of silver for one decigramme.

As the workable lead is a furnace product requiring a further treatment, in which a loss of silver also takes place, it is evident that the cupelling loss in this operation need not be taken into consideration. If, however, the per centage of silver is to be determined exactly, five decigrammes of such lead ought to be weighed, fused, and refined in two periods, and if, owing to too large a quantity of copper, the silver globule is not yet sufficiently pure, it is to be recupelled with one decigramme of proof lead. To the weight of the fine silver globule thus obtained, the amount of the cupelling loss, answering to the oxidized quantity of lead, is added, by which, and division of the sum by five, the real per centage of silver for one decigramme of the examined workable lead will be found.

If revived lead or bismuth is to be examined for silver, the former is cut into shreds like the workable lead, but the latter, on account of its brittleness, must be pulverized under the hammer. As no high per centage of silver can be expected in these compounds, an assay of five decigrammes ought generally to be employed. The weighed quantity of the alloy must either be immediately introduced, like workable lead, into a good cupel and fused, or in case the assay consists of a number of small particles, they should be first fused together on charcoal, and the globule removed from the support in two portions.

The separation of the argentiferous bismuth bead, which is to be subsequently cupelled from the oxide of bismuth formed during the oxidation, should be effected with great care, in order to prevent particles of the brittle material remaining. The globule ought never to be raised from the accumulated oxide, but the latter always detached by means of the pincers.

The pure globules obtained in the cupellation are to be measured in the scale, and the content of silver in one decigramme calculated from the weight found.

e. EXAMINATION OF METALLIC COMPOUNDS IN WHICH
ANTIMONY OR ZINC PREVAIL.

The subsequent observations apply particularly to argentiferous antimony or zinc.

If the antimony contains some per cents. of silver, it can be treated on charcoal in the oxidating flame ; the antimony volatilizes, and the silver remains as a dull bead. If, however, this does not happen, the silver may probably have been carried away by the current of air, and consequently the following operation must be performed.

Weigh one decigramme of the alloy and fuse it upon charcoal, in the reducing flame, with two decigrammes of proof lead, and a small quantity of borax, and subject the metallic button to the oxidating flame, to expel the antimonide of zinc. Oxidation of the lead does not commence until nearly the whole of these metals are volatilized. When the fumes cease, the blowing must be discontinued, the workable lead allowed to refrigerate, then separated from the scorix, and cupelled. This treatment is preferable for antimony, even when much silver is present, because, without an addition of lead, the last traces of antimony are expelled but imperfectly from the silver, and often a minimum of this metal might remain ; besides, if other metals, as copper or iron, are present, these are segregated by the lead in cupellation.

f. EXAMINATION OF METALLIC COMPOUNDS IN WHICH TIN
IS THE PRINCIPAL INGREDIENT.

Take for example Argentiferous tin.

As tin cannot be separated from silver, either by cupellation or volatilization, upon charcoal, it must be treated in the following manner :—

Weigh, of the substance to be examined, one decigramme, and dress it with—

Lead, 5 decigrammes ;

Soda, 50 milligrammes ;

Borax, 50 milligrammes.

Envelop the assay in a cornet of soda paper, place the whole in a prepared charcoal cavity, and heat in a strong reducing flame, until the metals form an orbicular alloy, and the borax and soda,—the latter is employed to prevent an oxidation of the tin,—are vitrified. When this is accomplished, the metallic globule alone is to be touched with the blue flame, care being taken to prevent a violent oxidation of the tin, so that the glass may imbibe all the oxide formed. If reduced particles of tin appear upon the sides of the scorice, discontinue the blowing, and allow the assay to cool. The refrigerated globule, to which one decigramme of borax is to be added, must now be treated upon another piece of charcoal, first with the reducing flame, and, if perfect fusion is effected in the oxidating flame, until it presents a bright surface. The workable lead, rendered by this operation free from tin, is submitted to oxidation and cupellation, and the per centage of silver ascertained upon the balance, or, if too small, upon the scale.

g. EXAMINATION OF METALLIC COMPOUNDS, THE PRINCIPAL
INGREDIENT OF WHICH IS MERCURY.

To this class belong native and artificial amalgams of silver, and argentiferous mercury.

One decigramme of the substance is weighed, placed in a small glass tube, with a bulb at one end, as in Fig. 44, page 202. The tube is held inclined, and heated gradually over a spirit-lamp. A very moderate heat volatilizes the greater part of the mercury, which deposits gradually, in a metallic state, upon the upper part of the tube. The bulb must now be heated to redness, and kept in this state until no more mercury is eliminated, when the tube is allowed to cool. After refrigeration, by slightly agitating the vessel, the globules of mercury readily cohere, and the main globule formed can be poured out, by gradually inverting the tube.

If the substance submitted to this operation was an amalgam, the silver remains in the bulb as a porous bead, and can readily be extracted. This globule must be fused in the reducing flame with borax, and one decigramme, or, if it contains copper, two to three decigrammes, of proof lead, and the formed workable lead treated in the usual way. The weight of the silver globule obtained is then ascertained upon the balance, and, if a native amalgam has been employed, the cupelling loss deducted.

However, if the distilled alloy be argentiferous mercury, the residuum in the tube is very inconsiderable, and cannot be detached from the bulb without great difficulty. In this case, the greater part of the tube must be cut off with a file, and what remains in the bulb dressed with one decigramme of lead, and 50 milligrammes of borax, and the whole placed in a charcoal cavity, and submitted to a strong reducing flame, until the metallic compound exudes from the glass. The argentiferous lead is, after cooling, easily separated from the glass and charcoal, and must be refined upon a well burned cupel of levigated bone ashes, and the resulting silver globule measured upon the scale.

The cupellation is necessary in both cases ; 1,—because the silver cannot be fused in the tube, and consequently all the mercury is not eliminated ; 2,—because the amalgam obtained from amalgamated ores often contains various metals, which remain in the distillation, and can only be separated from the silver by cupellation. If, however, gold be extant in the assay, it cannot be segregated in this way, but must be treated in the manner given under the examination for that metal.

A. EXAMINATION OF METALLIC COMPOUNDS HAVING IRON OR STEEL FOR A PRINCIPAL INGREDIENT.

As iron or steel cannot be united immediately with lead in the Blowpipe flame, their union must be effected in an indirect manner. It is generally known that sulphide of iron combines with lead, if these bodies are treated with borax in the reducing flame ; and ultimately, by heating in the oxidating flame, sulphur

volatilizes, and the iron oxidizes and is absorbed by the borax, either in the state of a protoxide or a sesquioxide. Therefore, if iron or steel is united with sulphur, the silver contained in the mineral may be separated as easily as if argentiferous sulphide of iron, *et cetera*, were assayed.

When hardened steel is to be examined for silver, it must first be submitted to a red heat, allowed to refrigerate, and, when cold, its surface cleansed, and about the necessary quantity for an examination taken off it with a file. One decigramme is then to be weighed and dressed with—

Sulphur, 50 milligrammes ;

Lead, 8 decigrammes ;

Borax, 1 decigramme.

The whole must then be enveloped in a soda paper cornet, placed in a cylindrical charcoal cavity, and heated in the reducing flame until the assay forms a molten bead. By this treatment the sulphur combines with the lead and iron.

As one decigramme of borax is not sufficient for imbibing the whole oxide of iron formed in the oxidation following the fusion, another decigramme of the flux is to be added, the whole re-fused, and submitted to a very strong oxidating flame until the impure lead begins to exude from the glass. The assay must now be held so that the lead is in contact with the flame,—the sulphur volatilizes, the iron oxidizes, and the formed oxide combines with the borax. After elimination of the sulphur, and segregation of the iron, the blowing is to be interrupted, and the lead, presenting a bright surface, and containing all the silver of the iron, allowed to refrigerate. If, when cold, it be of a whitish color, it must be treated like an ordinary workable lead, and the weight of the silver globule ascertained ; if, however, it be brittle, and of a blackish aspect, it should, before cupellation, *et cetera*, be submitted to a re-oxidation.

II.—THE GOLD ASSAY.

Gold can be separated in the dry way, like silver, from substances in combination.

As it is not susceptible of oxidation, even when treated with borax or lead in the outer flame, and sustains no loss in the cupellation, its per centage in ores, minerals, furnace products, *et cetera*, may be ascertained with the greatest exactness. The examination for gold, however, is more difficult than for silver, as, in most instances, gold, submitted to Blowpipe analysis, contains a quantity of the latter metal, which in native gold varies from 2 to 35 per cent.

From examinations of silver ores dressed in the Freyberg works for gold, the following results were obtained :—

Videlicet.—That all sulphides of iron holding from 0·0142 per cent. to 0·456 per cent. of silver, contain gold. This metal forms an ingredient also of the furnace products which are produced in the smelting of auriferous silver ores.

The various auriferous substances are brought, according to their nature and composition, under several heads ; thus :—

¶ A. Gold Ores, Auriferous Silver Ores, and Furnace Products containing Gold and Silver.

¶ B. Metallic Compounds, consisting of—

- a. Gold and Silver.
- b. Gold and Silver, alloyed with other metals.
- c. Gold and Mercury.

¶ A. *Examination of Gold Ores, Auriferous Silver Ores, and Furnace Products containing Gold and Silver.*

Under this class are ranged—

1. Native Tellurium, containing, according to KLAPROTH 0·25 per cent. gold, but no silver.

2. All ores and minerals, containing silver, and also gold ; as Graphic Tellurium,—Graphic Gold,—the composition of which is 60 tellurium, 30 gold, and 10 silver ;—according to BERZELIUS, it contains 52.0 tellurium, 24.0 gold, 11.3 silver, 1.5 lead. Yellow Tellurium,—Weiss-silvanerz, containing, according to KLAPROTH, 44.75 tellurium, 26.75 gold, 19.50 lead, 8.50 silver, and 0.50 sulphur ; Black Tellurium, composed of 32.2 tellurium, 54.0 lead, 9.0 gold, 0.5 silver, 1.3 copper, and 3.0 sulphur ; Noble Molybdena Glance, was found to contain 4.9 gold, and about 0.3 silver.

3. All argentiferous sulphides of Iron and Copper.

4. *Roh-* and *Blei-stein*.

5. The auriferous dross of gold- and silversmiths.

Of Gold ores, containing little or no silver, a quantity sufficient for one examination is prepared, and treated in the same manner as an assay of silver. After cupellation, the color of the globule must be observed, for if only 2 per cent. of silver were present, the gold would acquire a brass color. If the gold be pure, which is known from the color, its weight may be determined either upon the balance or the scale. When its color is very light, it it must be submitted to a new treatment, which will be hereafter described.

Of substances containing more silver than gold, a portion of 10 to 15 decigrammes must be reduced to powder, dressed and treated as in an examination for silver. From the quantity of silver yielded, the number of assays necessary for obtaining a sufficient quantity of gold, to make a quantitative determination, is calculated. Should the substance contain not more than 0.114 per cent. of silver, a considerable quantity of it ought to be pulverized, and at least 2.5 grammes taken ; on the contrary, if the per centage be larger, *exempli gratia*, 0.29 per cent., 10 to 15 decigrammes are sufficient for an examination. The dressing of the assay with lead and borax is effected in exactly the same manner as an argentiferous one, but when copper is present, the proportion of lead must be augmented. The fusion and cupellation can also be achieved in a similar way to the assaying of a silver alloy.

Sulphides or minerals containing large quantities of sulphur, and little silver, are analyzed in the following way:—24 to 36 decigrammes of the pulverized substance are divided into parcels of three decigrammes each, placed in a clay basin, the inside of which is covered with a coating of redde, and roasted without addition of charcoal, like a copper assay.

When nearly all the sulphurous vapor is eliminated, the assay is to be repowdered, and heated until no appreciable quantity of sulphur remains. This being accomplished, the residuum must be placed in a porcelain basin, over a lamp, and treated with a sufficient quantity of hydrochloric acid to dissolve out the formed oxide of iron or copper. The metallic oxides and the silver being dissolved, the gold, with some extraneous matters, will remain. The solution is then to be evaporated to dryness, and treated with boiling water. The soluble chlorides dissolve, and are to be separated from the chloride of silver, *et cetera*, by filtration and edulcoration. The filter holding the insoluble ingredients is to be dried, and after desiccation opened, and dressed cautiously with five decigrammes of lead and one decigramme of borax. The dressed assay must now be placed in a charcoal cavity, submitted to a feeble oxidating flame, in order to carbonize and consume the filter, and then treated with a strong reducing flame. In this operation the chloride of silver is decomposed, its base alloying with the lead and gold, the earthy constituents being dissolved in the borax. The metallic alloy is oxidized and cupelled in the ordinary way, and the auriferous silver globule obtained treated according to the described method for the separation of gold from silver.

It sometimes happens that sulphides containing less than 4 loths, = 0.114 per cent. of silver, are to be analyzed.—1 loth = 14.58808 grammes = 239.68 English grains.—In this case, 36 decigrammes are not sufficient, therefore, according to the percentage of the noble metal, the following proportions must be taken, *videlicet*:—Of a substance containing,—

3 loths = 0.0939 per cent. of silver, 48 decigrammes.

2	„	= 0.0616	„	„	72	„
1	„	= 0.0370	„	„	144	„
$\frac{1}{2}$	„	= 0.0142	„	„	288	„

As the weighing of a quantity of nearly 290 decigrammes in portions of three decigrammes each, would be very troublesome, it is sufficient to weigh upon an ordinary balance, according to the apothecaries' weight, of an ore containing,—

3	loths of silver in the cwt.	80	grains ;
2	" " "	120	" "
1	" " "	240	" and
$\frac{1}{2}$	" " "	280	" "

and to pulverize it well.

For the same reason, as it would be exceedingly inconvenient to roast a substance in parcels of three decigrammes each, the following expert method may be undertaken :—

Upon an iron foil, bent into a basin shape, place the whole assay, besmeared several times with clay water, and well dried ; and submit it to a strong red heat, continually stirring with a platinum spatula until no more sulphurous acid vapors are evolved, then triturate the residuum in a mortar, and subject the powder to a second roasting.

The assay, after these operations, must be introduced into a porcelain basin, placed over a spirit-lamp, treated with hydrochloric acid, evaporated to dryness, and then mixed with a quantity of water to dissolve out the soluble chlorides, the menstruum filtered, what remains on the filter edulcorated, desiccated, and, if it exceeds one decigramme, weighed in parcels of one decigramme each.

Each decigramme of this residue is to be dressed with

Lead, 5 decigrammes ;

Borax, 1 decigramme ;

fused like an argentiferous assay, and cupelled as already stated. The treatment of the auriferous globule will be described in the subsequent paragraphs.

¶ B. *Metallic Compounds.*

a. EXAMINATION OF SUCH AS CONTAIN SILVER AND GOLD ONLY.

To these belong native gold, alloys of gold and silver, and the argentiferous gold, or auriferous silver, obtained from the assayings of auriferous minerals and ores.

As, up to the present time, no process is known for separating gold from silver in the dry way, the moist way, termed in this instance *Quartation*, or *Refining of Gold and Silver*, must be employed.

For effecting the separation, the proportion of the gold to the silver should not exceed 1 to 3, as, if more gold be present, the solution will be performed either imperfectly, or not at all. When the gold is more than the above proportion, it so protects the silver, that even when hot fuming nitric acid is poured over the alloy, a very slight action only ensues. It is therefore indispensable to make a preparatory assay for determining the composition of the alloy, and if too small a portion of silver be present, add a quantity sufficient for producing the given standard.

Gold containing even no more than 2 per cent. of silver, is, as has been previously stated, of a brass color; if 30 per cent. of silver are present, it appears lightly brass colored, but when the quantity of silver amounts to 60 per cent., a yellow color is no longer perceptible. Therefore, from the intensity of the tinge in an auriferous alloy, conclusions as to its composition, and the necessary quantity of silver, can be drawn.

When native gold of a brass color is to be assayed, it is generally supposed that the per centage of silver associated with it is not high; in this case 20 to 30 milligrammes of the alloy are weighed, fused with $3 \times 20 = 60$, to $3 \times 30 = 90$ milligrammes of pure silver,—reduced from the chloride of silver,—and a small quantity of borax, in the reducing flame. Of a light colored auriferous body, 50 milligrammes are weighed for examination, and alloyed in the same manner with $50 \times 2 = 100$

milligrammes ; or, if the per centage of silver does not amount to 30 per cent., with 110 to 120 milligrammes of pure silver. In alloys presenting a bright white aspect, and composed of about 40 gold and 60 silver, the per centage of the latter metal cannot be previously known ; therefore one decigramme of the alloy should be fused on charcoal, with 60 to 80 milligrammes of pure silver, and a small addition of borax. In compounds of gold and silver obtained from the assaying of minerals or gold ores, gold is generally prevalent, necessitating their fusion with double or triple their weight of pure silver. On the other hand, the composition of alloys produced from roasted ores or sulphides, is nearly always in the proportion of 25 of gold to 75 of silver ; therefore, such compounds may be analyzed without an extra addition of the latter metal.

After weighing, according to the quality of the alloy, 20 to 100 milligrammes, and fusing upon charcoal with the necessary quantity of silver, the produced globule must be placed upon an anvil, laminated, reheated upon charcoal, introduced into a small porcelain crucible, supported over a lamp, treated with pure nitric acid, and covered with a watch glass to prevent an elimination of nitrous fumes. When the solution of silver is complete, the whole must be filtered and affused three or four times with hot distilled water, to remove the last traces of the nitrate of silver. The metallic gold remaining on the filter must be then dried, heated to redness in a platinum crucible, and weighed ; but should the quantity be too minute for the balance, it must be alloyed with lead, cupelled, and its weight determined upon the scale. One decigramme of lead will be sufficient for the dressing, and a small quantity of borax. The affining of the auriferous globule is often very difficult, owing to the proportion of lead present, but a little practice will greatly facilitate the assayer in his experiments. For the purpose of rendering the calculations of per centage of gold in alloys more easy, is annexed the following example :—If an alloy of 30 milligrammes has been assayed, and the product of gold obtained 25·5 milligrammes, we have,—

$$30 : 25\cdot5 :: 100 : x = 85 \text{ per cent. of gold.}$$

If in native gold, or in an artificial alloy, the per centage of silver is also to be ascertained, the assay must be cupelled, before fusion with fine silver, with one to two decigrammes of proof lead, to segregate any other metals, such as iron or copper; after affining, weighed; and the per centage of silver calculated from the difference, provided the quantity of gold has been ascertained.

b. EXAMINATION OF METALLIC COMPOUNDS, CONTAINING OTHER METALS BESIDES GOLD AND SILVER.

The most numerous of these are gold, alloyed with copper and silver.

Of this alloy, 30 to 50 milligrammes are to be weighed, fused according to the per centage of copper, with three to eight decigrammes of proof lead, and an addition of borax, upon charcoal, in a good reducing flame; then affined like a cupriferous workable lead. The copper oxidizes with the lead, the gold and silver remaining pure. If, however, after cupellation, the gold globule does not appear sufficiently pure, owing to a trace of copper, the bead ought to be immediately remelted with one decigramme of lead, and again affined upon a clear portion of the cupel.

The other observations agree with those described in the preceding pages.

c. EXAMINATION OF METALLIC COMPOUNDS CONSISTING OF GOLD AND MERCURY.

Although these compounds are not found in nature, they are produced in auriferous amalgamations. To effect their analysis, 50 milligrammes ought to be distilled in a glass tube, like amalgams of silver, and the residue cupellated with one decigramme of lead, and the refined globule weighed. If its color be very white, it is a proof of the presence of silver; and, therefore, it must be submitted to Quartation, which has been previously

described, and the quantity of gold and silver determined. Of poor amalgams of gold and silver, several samples of one decigramme should be introduced into a glass retort and distilled, and when the mercury is eliminated, new quantities of the amalgam ought to be added, and distilled, until the residue of argentiferous gold is sufficient for weighing. The operation being terminated, the gold globule is to be weighed, and, if necessary, fused with two to three parts of pure silver; treated with nitric acid; and the weight of the pure gold determined, from which the proportion of silver may be calculated.

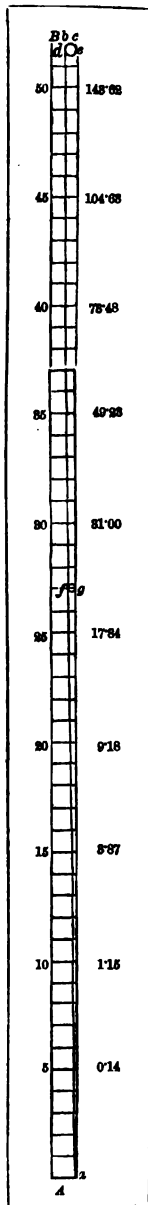
The weight of the gold and silver is to be divided by the number of decigrammes of mercury submitted to distillation, whereby the per centage of the two metals in one decigramme of the assay under examination is obtained.

1. *Determination of the Weight of Gold or Silver Globules by their Volume.*

It often happens that the affined globule obtained in quantitative Blowpipe analysis, from one decigramme of an argentine or auriferous substance, is so small that its weight cannot be determined exactly, even upon the most delicate balance; on the contrary, quantities sufficient to compensate for this poverty could not be operated upon, without depriving the Blowpipe examinations of their most important feature, *videlicet*—their simplicity and promptitude of execution. In order to avoid this difficulty, HARKORT endeavored to calculate the per centage of any assayed subject by measuring the diameter of the metallic globule obtained. Experience has proved the correctness of this method, notwithstanding the differences arising from inequalities in the form of the globules.

Harkort's Scale.—Fig. 47 represents a well polished ivory plate, upon which the line A B is drawn, parallel with one of its sides; then at a short distance from A B are two other lines, *a b* and *a c*, convergent at *a*, and forming an acute angle. The line A B is divided into equal sections by small parallel lines, which cross the convergent lines *a b*, *a c*, forming the bases of so many

Fig. 47.



triangles, similar to the large one, $b a c$; all these triangles possess a common angle, the sides opposite to which are parallel to one another, consequently a proportion between these homologous sides can be established: *exempli gratia*, between the large isosceles triangle, $b a c$, and the small one, $f a g$, there exists the proportion—

$$a b : a f :: b c, :: f g, \text{ and } :: a c : a g.$$

According to the above, the space comprised between the two lines $a b$ and $a c$ may serve for the determination of the volume of a sphere, and consequently of its weight.

If we suppose that a spherical metallic button be placed upon the scale between the lines $a b$, $a c$, and moved towards a , until the convergent lines from it became tangents, its diameter will be ascertained and expressed by the line uniting both tangent points. If, for instance, the button is found to correspond with the space between the two lines $d e$, its diameter is thus represented; and another globule, introduced into the scale, and touching the lines at $f g$, would have the diameter of this part.

The position of the two metallic globules being ascertained, and their distances from the point a established, by the divisions upon the line $A B$ with which they correspond, it is easily conceived how, from the relative proportions of their diameters, their weights may be ascertained. It will be as well here to remark, that the weight of homogeneous spheres is in direct proportion to that of their volumes, and the volumes are as the cubes of their diameters. Therefore, if L and l be the distances between the small globes and the point a ; V and v the two volumes, W and w the two weights, we have—

$$V : v :: (d e)^3 : (f g)^3 ;$$

and because

$$d e : f g :: a d : a f :: L : l$$

we have

$$V : v :: L^3 : l^3$$

and consequently, the proportion of the weights to the volumes—

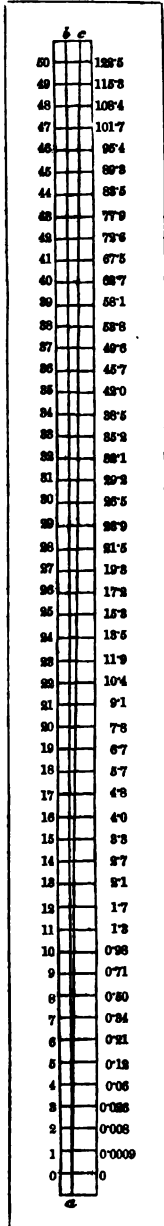
$$W : w :: L^3 : l^3$$

Though this proportion, indicating only the relation existing between W and w , gives no value to these quantities, it is evident that if the value of W be fixed, that of w is immediately established, according to the following equation :

$$w = \frac{W \times l^3}{L^3} \text{ or } = \frac{W}{L^3} \times l^3$$

In order to give a known value to W , the weighing of the button $d e$ is sufficient. HARKORT, therefore, selected an ore of a common per centage, and, having obtained equal results from the different globules, after repeatedly cupelling, he weighed the most regular, and found that its weight corresponded to a content of 154 loths in a hundredweight, or 4.354 per cent. This is the Saxon hundredweight=110 pounds, each of which is divided into 32 loths. As the German author graduated his scale according to this division, it is retained, care being taken to give also the expression in per cents., to avoid mistakes. He placed it upon the scale, and finding that it agreed exactly with the number 52 of the line $A B$, he inscribed there the weight indicated by the ball corresponding with this point, thus establishing a standard of comparison for smaller globules. The first point of the proposition being established in this manner, it is evident that by introducing the value of W into the formula, $w = \frac{W}{L^3} \times l^3$, the weight of any ball w may be determined. It is also clear that the constant factor $\frac{W}{L^3}$ remains the same in all the different values of w ; and therefore, the only care to be taken is, to determine the exact position of the globule upon the scale,—consequently, the distance l between it and the point a : for effecting this, the button is held between a pair of forceps, placed between the two lines on the scale, and

Fig. 48.



then moved until its tangent point is ascertained. In order to accomplish this exactly, a good magnifying glass is requisite; also a perpendicular direction from the eye to the scale.

Although in such an operation it is supposed that the globules are always orbicular, and of the same specific gravity, this is not the case, because, during the fusion, the globules are compressed by virtue of their gravity, and conserving their flat form after refrigeration; also the degree of density is dependent upon the time allowed for cooling. However, in the limits between which the scale may be employed, the influence of the temperature, as well as the form of the globule, are of no avail, as HARKORT has ascertained from numerous carefully conducted experiments.

PLATTNER'S Scale—its use in quantitative Silver Assayings. The examinations for silver, described in the preceding pages, being effected from one decigramme,—a standard representing the hundredweight in small operations,—PLATTNER constructed a scale, in order to use it for metallic globules obtained from an assay of this weight. He triturated, therefore, intimately, a quantity of *ruby silver*, with an equal quantity of a poor calcareous silver spar, and cupelled the mixture; this operation was repeated several times, and constantly gave 122.5 loths, or 5.48 per cent. silver. The assay was then submitted to the Blowpipe, and the same result obtained.

The annexed wood engraving—Fig. 48—

is a representation of this scale, divided into 50 parts, the first of which answers to the globule obtained from one decigramme of an ore containing $\underline{5.48}$ per cent. The constant factor $\frac{W}{L}$ 3.48 is therefore equal to 0.00098 for the division in loths; and $\frac{5.48}{50} = 0.0004384$, if the content be expressed decimally.

It is evident that a scale so constructed might be greatly extended, in order to serve for measuring metallic globules of a far greater volume than what is indicated in the foregoing remarks. This, however, is inconvenient; as the weight of spheres varies directly as the cubes of their diameters, the differences of weight for each division of the scale increase considerably,—therefore, a button, having a greater gravity than can be ascertained upon any scale, may readily be weighed upon the balance.

The divergence of the two lines $a\ b$ and $a\ c$ requires a remark. The smaller the divergence is, in general, the less will be the variation in weight for each descending line, and the more accurately the diameters of globules can be compared; but this is again limited in practice, for if the divergence be so minute that the difference between the diameters of two globules cannot be ascertained, the scale becomes useless. The most convenient divergence has been found to be two-sevenths of a line, in a length of fifty English lines.—The English line is equal to 3.17494 millimetres.

The value of the scale is considerably increased by marking at the side of each division the per centage indicated in a metallic globule corresponding to it. For the smallest of these three decimals are sufficient; if, however, the next decimal number exceed 5, the preceding may be increased by 1. A silver globule, when placed upon the scale, is often found not to coincide with any of the perpendicular lines: in this case, the distance of the touching point from the upper line is ascertained; then the difference of weight corresponding to the divisions enclosing the globule is multiplied into the known fraction, and the product added to the weight indicated by the under line. Thus if a silver globule, placed at two-thirds of the distance between 43 and 44, corresponds to a richness of silver comprised between

83.48 and 77.916 loths, or, in decimals, 2.37160 and 2.21545 per cent., the difference of the centesimal value being 0.15615,—this, multiplied by two-thirds, gives 0.10410, which, added to 2.21545, shows the product to be 2.31955 per cent. The exactness of these calculations depends chiefly on the skill exercised in placing the smallest globules upon the scale, to ascertain their diameters. The operator may, however, control his results, by weighing a number of globules obtained from an ore of a mean value, in one lot, upon the balance, after measuring them singly upon the scale, and dividing the whole weight by the number of globules taken.

2. *Application of the Scale for determining the weights of Gold Globules, obtained in Quantitative Assayings.*

It is very easy to comprehend, that upon such a scale as the one just described, small gold globules obtained in cupellation may also have their diameters and weights determined. If gold globules were as much compressed in refining as silver, their contents might be ascertained from the relative specific gravity of the two metals, but the cohesion of molten gold, being much greater than that of silver, prevents it from assuming the flat form that generally accompanies the latter metal. It was therefore necessary to calculate a new factor for W. This, PLATTNER effected, by dissolving 946 milligrammes of fine gold in nitrohydrochloric acid,—aqua regia,—and precipitating the metal with protosulphate of iron, then filtering, edulcorating, desiccating, and igniting the residue. The protosulphate in this instance, is converted into sesquisulphate, and sesquichloride of iron.—The metallic gold was next mixed with 14.544 grammes of calcareous spar, and portions of this submitted to cupellation, and also to the Blowpipe assaying. The mixture, 15.5 grammes, contained 946 milligrammes of gold, or 6.103 per cent.

The metallic buttons obtained in the cupellation agreed very closely with the above, each weighing 6.09,—and the globules resulting from the analysis with the Blowpipe weighed, altogether,

30.5, indicating a mean gravity of 6.08 for each, corresponding to 214.5 loths in a cwt.

The experiments agreeing, PLATTNER placed one of the globules between the lines *a b* and *a c* upon the silver scale, and found its tangent points to be in the middle, between the numbers 46 and 47. Consequently, the fact that a gold globule placed upon this part weighed 6.07 milligrammes, = 214.5 loths per cent., establishes the subjoined general equation :—

$$w = \frac{W}{L} p$$

$$W = 6.07, \text{ or } 214.5 \text{ loths.}$$

$$L = 46.5 \quad \quad \quad \text{,,} \quad \quad \text{,,}$$

<i>Divisions of the Scale.</i>	SILVER.		GOLD.	
	<i>Loths per cwt.</i>	<i>Per Cents.</i>	<i>Loths per cwt.</i>	<i>Per Cents.</i>
1	0·00098	0·000028	0·00213	0·00006
2	0·00184	0·000228	0·01708	0·00048
3	0·02646	0·000752	0·05760	0·00164
4	0·06272	0·00178	0·13658	0·00388
5	0·12250	0·00348	0·26667	0·00757
6	0·21168	0·00601	0·46081	0·01309
7	0·33614	0·00955	0·73174	0·02079
8	0·50176	0·01425	1·09229	0·03103
9	0·71442	0·02029	1·55523	0·04418
10	0·98000	0·02784	2·13388	0·06061
11	1·30438	0·03705	2·83952	0·08066
12	1·69344	0·04811	3·68648	0·10473
13	2·15806	0·06116	4·68703	0·13296
14	2·68912	0·07639	5·86399	0·16630
15	3·30750	0·09396	7·20015	0·20455
16	4·01408	0·11404	8·73832	0·24824
17	4·81474	0·13678	10·48129	0·29776
18	5·71536	0·16237	12·44187	0·35346
19	6·72182	0·19096	14·63285	0·41570
20	7·84000	0·22273	17·06704	0·48485
21	9·07578	0·25783	19·75936	0·56134
22	10·43504	0·29644	22·71623	0·64534
23	11·92366	0·33874	25·95683	0·73741
24	13·54752	0·38487	29·49184	0·83784
25	15·31250	0·43501	33·33406	0·94699
26	17·22448	0·48933	37·49628	1·06523
27	19·28934	0·54799	41·99131	
28	21·51296	0·61116	46·83195	
29	23·90122	0·67903	52·03100	
30	26·46000	0·75170	57·60126	
31	29·19518	0·82941	63·55552	
32	32·11264	0·91229	69·90659	
33	35·21826	1·00052	76·66727	
34	38·51792	1·09426	83·85036	
35	42·01750	1·19368	91·46866	
36	45·72288	1·29894	99·53497	
37	49·63994	1·41022	108·06209	
38	53·77456	1·52769	117·06282	
39	58·13262	1·65149	126·54996	
40	62·72000	1·78182	136·53632	
41	67·54258	1·91882	147·03468	
42	72·60624	2·06268	158·05785	
43	77·91686	2·21595	169·61864	
44	83·48032	2·37160	181·72984	
45	89·30250	2·53700	194·40425	
46	95·38928	2·70992	207·65467	
47	101·74654	2·89053	221·49391	
48	108·38016	3·07898	235·93476	
49	115·29602	3·27545	250·99002	
50	122·50000	3·48011	266·67250	

III.—THE COPPER ASSAY.

The composition of natural and artificial substances containing copper, is always to be considered before the commencement of the operation, as upon this the difficulty of the examination depends. For the same reason, cupriferous substances are to be divided into the classes of,—

¶ A. Ores, Minerals, and Furnace Products :—

- a. Containing volatile ingredients ;
- b. Containing the Copper in an oxidized state, with or without acids and water, vitrified, or washed in another manner with earthy constituents.

¶ B. Metallic Compounds, the Copper of which is either a principal or an accidental constituent :—

- a. Plombiferous Copper, and Cupriferous Lead ;
- b. Alloys of Copper with iron, nickel, cobalt, zinc, and bismuth, one or several of these metals, often also with lead, antimony, and arsenic, as accidental constituents ;
- c. Stibiferous Copper ;
- d. Stanniferous Copper.

For the ores, minerals, and furnace products belonging to the first class, roasting is necessary, previously to the fusion of the copper, in order to eliminate sulphur and arsenic ; but it is not so with those of the second class. The impossibility of producing pure copper immediately from ores, minerals, and furnace products, containing several volatile metals, many of which cannot be expelled by roasting, is well known from the fusion of copper ores on a large scale, as well as from the common test in the dry way. As it may be necessary to give particulars concerning this point, the manipulation for obtaining

the copper, as correctly as possible, by the aid of the Blowpipe, will at one be proceeded with.

¶ A. *Ores, Minerals, Products of Furnaces and Manufactures.*

a. EXAMINATION OF SUCH AS CONTAIN VOLATILE INGREDIENTS,
SULPHUR AND ARSENIC.

To this class belong all copper ores prepared on a large scale ; of minerals,—Kupfer Glanz, Purple Copper, Tennantite, Grey Copper, common sulphides of copper and tin, Bournonite, Melanglanz, *et cetera* ; and of furnace products, *kupferstein, kupferleg, cupriferous rohstein, bleistein, tutty, et cetera*.

Of these substances, the necessary ground ore is prepared, and one decigramme weighed for examination. As they must be roasted previously to the fusion, the sample is mixed in the agate mortar, either with three times its volume of dry pulverized charcoal, or with 20 to 25 milligrammes of graphite, which in most cases, particularly for substances containing much arsenic, is more advantageous than charcoal. It is then placed upon a clay basin, and besmeared with red ochre—sesquioxide of iron.

A charcoal prism of sufficient size is introduced into the charcoal holder, and tightened ; a cavity is then made in it, and from its contiguous sides so much cut out with a knife as is prescribed by the orifice *b*, Fig. 27, page 27, in order to allow the flame access to the cavity. The platinum foil and wire is placed in it, the clay basin, taken with the pincers by the rim, is sunk in a horizontal position, until it reposes upon the platinum wire. For obtaining a horizontal position of the basin, it is necessary to hold the pincers so that their arms can pass through the orifice. After these preparations, the Blowpipe is provided with a point of medium size, then an oxidating flame, not too strong, is directed through the orifice upon the open space below the basin.

In order to produce a red heat on the surrounding space, as well as on the basin, the charcoal holder should be at the dis-

tance of an inch and a half from the wick of the lamp, for if it is too near the flame, sufficient heat is not produced ; whereas, at a greater distance, by the introduction of a large supply of heated air into the cavity, the combustion of the charcoal is too much accelerated, generally impairing the operation. The basin is kept for some minutes in a state of redness, though care must be taken to blow moderately, in order to prevent a partial or perfect fusion of the sample. In this operation, the presence or absence of volatile ingredients may be detected from the smell of the eliminated gases. Further ; the charcoal, added to the ore, is wholly destroyed, which can be ascertained by examination with the iron spatula, heated over the free flame of the lamp, in order to prevent the adhesion of small particles of the substance. The basin is removed from the holder, its contents placed in the agate mortar, and reduced to powder. Generally the ore changes its color in the first roasting, forming a porous mass, which may be easily detached from the basin, and seldom requires the use of the spatula.

In this roasting, which is terminated in about ten minutes, the greater part of the volatile ingredients is removed—sulphur, arsenic, and antimony—the addition of charcoal preventing the formation of sulphates and arseniates. For the complete elimination of the volatile substances preventing the reduction of the copper, a second roasting is necessary. The residuum of the first roasting is again mixed with three times its volume of pulverized charcoal, the basin, prepared as before, placed on the holder ; if the charcoal, mixed with the substance, is in full incandescence, a somewhat stronger heat is applied, and as soon as half the charcoal is consumed, the assay is examined for volatile ingredients. If no smell indicating sulphur or arsenic is perceived, the remainder of the charcoal is allowed to burn under continued insufflation, after which the roasting is considered to be completely finished. On the other hand, if the volatile constituents are not wholly eliminated, a third roasting is necessary. It very seldom occurs, except in substances containing much arsenical nickel, that a fourth roasting is required ; two are generally sufficient. In order to learn when the roasting is finished, it is

better to weigh the basin several times successively, as after the assaying its weight remains constant ; this, however, is difficult, and occupies too much time ; therefore the smell exhaling from the roasted substance usually serves for ascertaining this period.

Regarding the richness of substances roasted with pulverized charcoal, conclusions may be derived with sufficient certainty, from the color of the sample. The blacker the color, the richer it is in copper ; if reddish or whitish, it is poor. If, instead of charcoal, graphite is employed, the assay is kept at a red heat, until the gases disengaged become inodorous. The graphite being destroyed slower than charcoal, and consequently remaining longer in immediate contact with the substance, reduces continuously the volatile ingredients ; preventing, at the same time, the formation of sulphates or arseniates. As soon as the smell of the escaping gas ceases, the basin is taken from the charcoal, and the metallic substance, mixed with the remainder of the graphite, is triturated again in the agate mortar. This operation is indispensable, as, generally, during the roasting, the upper is sooner consumed than the lower portion of the graphite, in which unaltered metallic particles might remain, and these are again brought into contact with new portions of graphite. The sample is respread on the clay basin, and submitted to a new roasting in a higher temperature. At the commencement of this second operation, the smell of volatile matter is often perceived for some moments, indicating the volatilization of the remaining traces of arsenic and sulphur. The blowing is continued until nearly all the graphite is destroyed ; then the basin is removed from the charcoal.

Absence of smell from an assay roasted with graphite, is a sufficiently certain sign of the termination of the process. The constancy of the weight cannot be taken as a characteristic as the consumption of the whole graphite is not necessary. The possibility of adding a surplus of graphite to an assay, is the chief advantage of its employment in roastings. It is necessary to employ it in a pure state. If a cupriferous mineral, prepared on the large scale, contains heavy spar or gypsum, the sulphuric acid cannot be driven off by roasting ; though the heavy spar is

reduced, in a red heat, to sulphide of barium, and the gypsum to sulphide of calcium, both substances re-oxidize after the combustion of the charcoal. For the reduction of the copper contained in one decigramme of the substance, roasted in the manner here described, the following dressing was adopted, *videlicet* :—

Soda, 100 milligrammes ;

Borax, 50 milligrammes.

The soda reduces the oxide of copper, and other easily reducible metallic oxides ; and the borax dissolves the oxides of iron manganese, cobalt, and the earthy constituents.

The roasted substance, mixed with the soda and borax in the agate mortar, are enveloped in a soda paper cornet, and pressed into a cavity, prepared on the diametrical section of a good piece of charcoal, near one of its corners.

These preparations being effected, a pure and strong reducing flame is deflected directly upon the sample, until the paper, the substance roasted, and the graphite are destroyed, and metallic copper is seen floating, either on the scorise,—with its peculiar greenish-blue color,—or separately near them, in the form of a metallic globule.

In rich substances the reduced copper speedily appears, but more slowly, and sometimes not at all, if the ore is poor. Sometimes, after a long continued strong insufflation, the copper unites to one globule. It, however occurs oftener, that minute metallic particles remain in the scorise. It is therefore preferable to interrupt the process of reduction, so soon as the melted copper appears in the perfectly glossy scorise ; to withdraw the scorise cautiously from the charcoal,—if not colored red, which indicates vitrified sub-oxide of copper ; reduce it to fragments between folded paper, upon the anvil ; detach the visible globules ; levigate and lixiviate the mass in a porcelain basin, with a view to remove dross and charcoal ; and desiccate the basin with the copper, over the lamp flame. If the scorise are not completely separated, they remain united with the copper, and adhere to the basin, from which they must be detached by the spatula. In this case, it is necessary to re-levigate with water, and to desiccate a second time, after its affusion, when the copper may be easily detached. If the copper

reduced in this way, and examined by a microscope, displays its natural color, and is easily detached upon the anvil, its quantity may immediately be determined ; should it, however, appear of a grey color, fragile, or too ductile, these are signs of its contamination with other reduced metals. In this case, it ought to be fused on charcoal in the reducing flame, with 50 milligrammes of lead, and a small addition of soda and borax, until the copper unites with the lead, in the form of a bright globule, and the borax combines with the soda, as a limpid bead.

After refrigeration, the cupriferous bead is detached from the decrements, and affined according to the subsequent prescription. If, in the reduction of copper from a roasted substance, easily fusible metallic globules appear, which can be readily united to a greater one, it may be concluded—

1. That the roasting has not been entirely performed ; or that heavy spar or gypsum is present, the sulphur combining with the copper as sulphide, surrounding the pure copper, and forming with it a single button :

2. That the substance contains other metals, making an easily fusible alloy with copper.

As, by levigation and lixiviation, in the first case, a considerable loss will be sustained, owing to the inferior specific gravity of the sulphide of copper and its divisibility, another process for separating the sulphide, together with the pure copper, is to be pursued. Therefore, when these globules appear, the insufflation is interrupted, to ascertain their color. A black tinge indicates the presence of sulphur, whereas a greyish or whitish one proves the presence of other metals. Then the reduction is continued until all the copper is reduced into a ball, which may be effected by bringing the main globule successively in contact with all parts of the scorixæ. The reduction being completed, the ball is watched until it congeals, then quickly removed from the fluid scorixæ by the forceps, and allowed to cool. Should the earthy sulphate in the treated substance possess but half the weight of the whole, the metallic copper forms only a button, enveloped in a thick crust of sulphide. But if a small quantity only of earthy sulphate be present, the *kupferstein* forms a thin coating. The result is the

same if the roasting be imperfect, or if sulphur is found in an acid state. The globule, removed from the scorise, is fused on charcoal in the reducing flame, with twice its weight of lead, and a little borax, until all the sulphur of the cupriferous lead is volatilized, and a bright metallic surface appears. After refrigeration the metallic bead is detached from the colorless glass, and affined in the manner described hereafter. In this operation, if other metals are extant, these are separated at the same time.

In the second case, if the substance contains, besides copper, other metals oxidized in the roasting, it is also necessary to continue the fusion until the reducible metals form a single globule, and the recrementitious matters are fused. After the termination of the process, the scorise, with the metallic globule, are allowed to cool, and cautiously broken upon the anvil to detach all extraneous matters; as the alloy, in the reduction, rises above these scorise, and can easily be united to one ball, it is evident that no pulverization and lixiviation are necessary. Should antimony, tin, or bismuth be present, the globule is brittle; ductile, however, with lead. With a view to avoid pulverization and lixiviation for compounds difficultly combining in one globule, it is only necessary to add 30 to 50 milligrammes—according to the percentage of copper,—of granulated proof lead, by which means an easily fusible metallic globule is produced, which is to be affined in the manner subsequently described; the affining, however, occupies less time than the lixiviation.

b. EXAMINATION OF SUBSTANCES CONTAINING THE COPPER IN AN OXIDIZED STATE,

Either pure, or combined with Acids and Water, or vitrified with Earthy Constituents, et cetera.

Minerals.—Red Copper Ore, Cupriferous Manganese, Black Copper, Chloride of Copper, Azure Copper Ore, Malachite, Arseniate of Copper, Phosphate of Copper, Sulphate of Copper, *et cetera*.
Furnace Products.—All varieties of copper scorise; products of

manufactures, particularly the pigments prepared from copper and its oxides, and the cupriferos vitriols.

The substances are not roasted, and, with the exception of the vitriols, are treated as follows ; *videlicet*—

Substance, 1 decigramme ;

Soda, 1 decigramme ;

Borax, 50 milligrammes ;

if the copper exceeds 38 per cent., from 10 to 20 milligrammes of oxide of antimony, or 50 milligrammes of proof lead, are added.

The sample is mixed in the agate mortar, placed in the soda paper cornet, and introduced into a cylindrical cavity prepared on charcoal. The reduction of the copper is effected in the same manner as the assays resulting from previously roasted substances ; the soda, acting as the flux, combining with chlorine, as well as with phosphoric acid ; whereas, the borax dissolves the earthy particles, and the more difficultly reducible metallic oxides, and prevents the absorption of the soda by the charcoal, when chlorine and phosphoric acid are absent.

By the addition of antimony and pure lead, considerable losses, resulting from the spattering of the fine copper, often exceeding three per cent., will be avoided ; though it is certain that the evitiation of this inconvenience produces another one, namely, the remaining of a small proportion of foreign metals ; this, however, is unimportant, as the final purification is one of the most simple and easy operations.

For the examination of substances containing upwards of thirty per cent. of copper, or where the presence of lead is surmised, the oxide of antimony is not to be employed. In the first place, it is to be feared that the copper, forming a very small globule, might be driven off mechanically, should the antimony be volatilized. In the second place, an affinage with boracic acid would be unavoidable. The sulphates of copper cannot be treated in the manner already described, for, though the sulphuric acid is decomposed, and its sulphur partially combines with the sodium in the commencement of the operation, yet the former, owing to its great affinity for copper, separates again from the radical of the

soda in a continued reduction, forming sulphide of copper, which can with difficulty be rendered pure, and seldom without loss. It is necessary, therefore, to dissolve one decigramme of the sulphate in water, over the flame of a lamp ; to precipitate the oxide from the boiled solution by caustic potassa ; to filter, edulcorate, and desiccate the filter in a porcelain basin ; to burn the dry filter over a mortar ; to mix the ashes and metallic oxides with 50 milligrammes of soda, and 30 milligrammes of borax ; and to fuse the sample, enveloped in a soda paper cornet, on charcoal in the reducing flame. By this operation, the copper is immediately obtained pure, if the original substance contained no foreign metals. If, in this case, the levigation of the scorix should be avoided, 20 to 30 milligrammes of proof lead are to be added before the reduction, to form an easily fusible plumbiferous alloy, which can readily be affined by means of boracic acid.

¶ B. *Metallic Compounds.*

The copper can be separated with the Blowpipe from its compounds with other metals only by oxidation, which, acting upon those metals, partly volatilizes, and partly converts them into scorix ; whereas, the copper remains in a metallic state. Though this operation could be effected by a process analogous to the affinage of copper in smelting works, it is, however, difficult and always liable to loss of the metal. The following process will answer best in docimastic designs. It is founded upon the property that boracic acid possesses of dissolving, in the reducing, as well as in the oxidating flame, several metallic oxides,—particularly that of lead,—and of maintaining this combination, if it is not too much saturated, even in the strongest reducing flame ; so that, whatever the proportions of the copper to the lead may be, the separation can always be effected without the slightest loss to the former, and if a small portion oxidizes, it is reduced immediately. If the copper contains other easily oxidizable metals, besides the lead, these are all converted to oxides. It is therefore

evident, that alloys containing copper, lead, and such metals may be affined with the aid of boracic acid. Alloys of copper with antimony or tin require another treatment.

a. EXAMINATION OF ALLOYS OF COPPER AND LEAD.

The plombiferous copper, obtained by reduction of plombiferous copper ores.—The cupriferous lead, produced by reduction of cupriferous lead ores.—The cupriferous workable lead, produced on a large scale ; *Frischstücke, Saigerdörnern, Darrlinge, et cetera.*

Alloys of copper and lead, obtained generally in one globule, from the reduction of one decigramme of a substance, are separated as follows, *videlicet* :—

In a cavity, prepared on the diagonal section of a piece of charcoal, near one of its corners, a quantity of vitrified boracic acid, equal in weight to the plombiferous copper, is fused to a bead, the metallic globule placed near it, and both covered with a good reducing flame. As soon as the boracic acid is fused, and the metallic globule presents a shining surface, the point of the Blowpipe, the opening of which ought not to be too large, is advanced in the flame, in order to change, gradually, the reducing flame to a blue one, which is so directed upon the fused boracic acid, that the alloy remains untouched. It must also be well observed, that the metal remains in contact on one side with the glass, and on the other with the charcoal, as, even on the slightest inclination of the latter to the wrong side, the globule escapes under the glass, thus causing an interruption of the process. In this case it is necessary to incline the charcoal to the other side, under a feeble insufflation, to allow access of air to the globule.

While the glass is treated with the blue flame, the lead bound in the alloy absorbs oxygen from the atmosphere ; the resulting oxide is carried to the boracic acid by the motion of its surface, and instantly dissolved.

This process is continued until nearly all the lead is oxidized, and the metallic globule seems to assume a greenish-blue color ;

then a broader flame is directed upon the glass, to render the oxidation of the remaining lead slower, and to prevent a spattering of the copper. This inconvenience it is almost impossible to avoid, if the operation is performed in a groove of insufficient depth; therefore, the cavity ought always to have at least three-sixteenths of an inch in depth, and three-eighths of an inch in width. As soon as the greenish-blue color, peculiar to fused copper, and indicating the desired degree of purity, is obtained, the process is interrupted, the solid globule removed from the still fluid scorix, and its properties examined after cooling. The best indications of its perfect purity, are, the common red color of copper, perfect malleability, and arborescent appearance upon the surface of the broken globule, when viewed through the microscope. This last sign is only perceptible in globules resulting from an alloy of high per centage. If the scorix remaining upon the charcoal are of a yellow color, and limpid, the examination has been performed without a chemical loss of copper, and the bulb may be weighed. If the scorix appear reddish, or perfectly red, a loss of copper is indicated, which can be recovered in the following manner:

If, as already mentioned, the boracic acid is not over saturated with oxide of lead, the oxide and suboxide of copper may be easily reduced, and separated in a metallic state by a good reducing flame. The glass treated after the detachment of the affined copper globule, with the reducing flame, becomes limpid, and of a yellowish color, after refrigeration; the reduced copper being scattered through it in small globules. These globules are obtained either by fusing again the great copper globule, in a good reducing flame, allowing it to float in the scorix, covered by the flame, until all the small globules are united with it, and detaching it from the scorix as before; or by breaking the scorix enveloped in paper upon the anvil, and removing them by levigation and lixiviation, in a porcelain basin. In the first instance, the copper can be weighed immediately; whereas, the globules remaining after removal of the scorix ought to be dried in a basin over the lamp flame, and their weight ascertained, together with the large one.

From a glass containing a quantity of oxide of lead, besides

the oxides and suboxides of copper, a single globule of lead and copper is obtained by reduction. For removing the lead from this alloy, it is only necessary to fuse the ball for some moments with a small addition of boracic acid in another cavity on charcoal. With the exercise of due caution, however, these labors are very seldom required, as, generally, the copper is obtained pure and without loss.

If the substance examined for copper in this way contains also a quantity of silver,—determined by an examination for this metal,—it must be deducted from that of the obtained copper ; if, however, the per centage of silver has not been determined previously, it ought to be treated as an assay of silver, with fifteen times its weight of proof lead, and submitted to oxidation and cupellation, by which means the quantity of the former metal is determined.

If, instead of plumbiferous copper, as in the preceding case, cupriferous lead,—as it is sometimes obtained by the treatment of cupriferous lead ores—is examined, the alloy cannot immediately be affined, owing to the time necessary for oxidation ; in this case, the operation ought to be effected in two periods,—

a. The concentration, and

β. The affinage of the copper.

Although the concentration and affinage of a cupriferous alloy are performed with boracic acid in the manner already described, a quantity of the latter sufficient for the solution of all the oxide of lead cannot be added, because the remaining copper globule will seldom be obtained pure, owing to a tendency to imbed itself among the scorizæ ; therefore, such assays must always be concentrated previously to the affinage. The cupriferous lead is weighed exactly, fused to a globule with a small addition of soda and borax on charcoal in the reducing flame ; it is then treated with its weight of vitrified boracic acid, as though it were to be affined, until reduced globules of lead appear on the surface of the glass. The greater portion of the lead being oxidized in this way, and separated from the copper, the insufflation is suspended, and after cooling the metal in which the copper is now concentrated, it is detached from the scorizæ. If the glass appears like

enamel, which is generally the case, the operation is performed without loss of copper. By the concentration, a plombiferous globule is obtained, which may be treated as already described.

b. EXAMINATION OF ALLOYS CONSISTING OF COPPER, IRON, NICKEL, COBALT, ZINC, AND BISMUTH,

And in which the Copper is combined with one or several of these metals, and frequently with Lead, Antimony, and Arsenic.

To this class belong :—

α. The impure black copper obtained from the treatment of cupriferous lead ores on a great scale ; and the *Saiger-dörnern* ;

β. The black copper obtained on a large scale from sulphide of copper, copper slate, and other copper ores containing no lead ;

γ. The German silver, or argentan, and such compounds of copper with nickel as contain little or no lead.

α. Affinage of the impure Black Copper prepared on a large scale from cupriferous Lead Ores.

This species of black copper contains, besides lead and copper, iron, nickel, antimony, zinc, arsenic, and various other metallic constituents. It is very brittle, and can be reduced to easily segregating laminæ by percussion only.

One decigramme of the substance is weighed, fused on charcoal to a single ball, if not already consisting of one piece, and treated with one decigramme of boracic acid, as already described, until the globule is covered with a coating of oxides, and has lost its fusibility. In this process, lead, iron, antimony, zinc, arsenic, and other easily oxidizable metals, and a portion of the nickel, become oxidized. The formed oxides partly combine with the boracic acid, and partly volatilize. A portion of the nickel remains with the copper, in the form of a thin layer of oxide, greatly obviating

the affinage. Though, by a prolonged oxidation, this coating, as well as the rest of the nickel, are dissolved by the scorïæ, this cannot be effected without a considerable loss of copper. It is, therefore, better to add a quantity of pure lead, equal to the weight of the alloy, and to submit it to a new oxidation, perfectly equal to the first one. By this the nickel is oxidized, together with the lead, without loss of copper. Should the latter, notwithstanding these precautions, be dissolved in the scorïæ, these ought to be submitted to a reduction, and after levigation and lixiviation weighed, together with the main globule. The oxidized nickel is only reduced in a continued strong inner flame. As this sort of copper often contains half a per cent. of silver, the per centage of this metal ought to be determined, and deducted from the weight of the copper.

β. Affinage of the Black Copper obtained by the treatment on a large scale of Sulphide of Copper, Copper Slate, and other non-plombiferous Ores in general.

The copper in these alloys is generally in combination with either iron or zinc, or both. Brass also belongs to this class, although its per centage of zinc is large.

The metallic compounds of this order can be affined in the same manner as the plombiferous copper:—one decigramme of the substance is reduced to fragments, fused on charcoal, with one decigramme of proof lead, according to the per centage of zinc and iron, and a small quantity of soda and borax; after refrigeration and detaching of the scorïæ, the globule is treated with boracic acid, like plombiferous copper; the iron and a portion of the zinc oxidize, another portion of the latter volatilizes, the copper remaining pure. If a small portion of the copper is also oxidized, it can be recovered by reduction and levigation. The per centage of silver in these alloys is seldom so great as to be taken into consideration.

γ. Determination of the Per Centage of Copper in German Silver, and other Nickeliferous Compounds containing little or no Lead.

Nickel, in combination with easily fusible metals, oxidizes with difficulty, its oxide dissolving in boracic acid. If, however, the glass saturated by it is treated with the reducing flame, its greater portion will again be separated in the metallic state. Therefore, in the separation of nickel from copper by means of boracic acid and pure lead, the greatest care should be taken to prevent an oxidation of the latter metal, it being impossible to render it free from nickel in a reduction.

As a loss of copper will be sustained if an alloy contains upwards of 20 per cent. of nickel, it is necessary to examine such substances in samples of about 50 milligrammes; or 45 to 50 milligrammes of the substance are weighed, and fused with two decigrammes of proof lead, and some borax, on charcoal, in the reducing flame; if the alloy contains zinc, it is volatilized in this operation. After cooling, the borax glass, which, if iron or cobalt were present, would have the coloring from those metals, is detached from the metallic globule, and the latter treated with one decigramme of vitrified boracic acid, until the lead, with the greater portion of the nickel, is oxidized and imbibed by the scoræ; the remaining globule requires a higher temperature for continuing in fusion. This operation must be performed by means of the blue flame directed upon the scoræ, in order to allow the oxidation and scorification of the nickel. The metallic ball is to be kept in contact on one side with the scoræ, on the other with the charcoal, for if the point of the blue flame only acts upon the sample, the copper will oxidize;—the flame ought to be so directed that it spreads over it.

So soon as the scoræ are saturated with lead and nickel,—indicated by the reduction of a portion of the lead, and the difficulty with which the ball is kept in fusion,—the button is allowed to refrigerate, and is then removed from the scoræ by the pincers. This ball, containing but a minute portion of nickel, is treated with its volume of proof lead and boracic acid on charcoal, as already described. From an alloy poor in nickel, the copper is

obtained pure by this operation ; on the other hand, if the portion of nickel be considerable, the globule obtained is still nickeliferous. In this case, it is fused again with its volume of proof lead, and one decigramme of boracic acid ; and this operation ought to be repeated with twice the quantity of lead, and one decigramme of boracic acid, if, after the second oxidation, the copper has not lost its white color.

The scorizæ formed in these operations must be of a red color, from the oxide of nickel, and transparent when compressed with the pincers. Green colored transparent scorizæ indicate dissolved oxide of copper ; a brownish tinge, with opacity of the scorizæ, indicates the presence of dissolved suboxide of copper : these colorings, however, only appear when the affined copper is kept fusing too long. Scorizæ of a brownish-red color ought to be covered with the reducing flame after removing the globule, to reduce the copper ; and then obtain it by levigation and lixiviation. The reduction should be continued until the red coloring of the scorizæ has disappeared ; on the contrary, if the scorizæ are colored blue by the oxide of copper, it is difficult to deoxidize it, even in the best reducing flame. The copper obtained from this species of scorizæ is generally nickeliferous, and seldom exceeds 0·1 milligramme.

If, after these operations, the copper has attained its natural color, its weight is ascertained, in order to calculate the value of the treated alloy. The following examples show that, notwithstanding all precautions, a small loss of copper is sustained in the described treatment ; this takes place in the affinage, as well as in the first and second treatment with boracic acid, though it cannot be perceived, owing to the dark color of the scorizæ :—

1. From 50 milligrammes of an alloy of 50 copper, 25 nickel, and 25 zinc, 24·5 milligrammes of pure copper were obtained. In this instance, therefore, there was a loss of 0·5 milligramme = 1 per cent.

2. A mixture of 40 milligrammes of granulated nickel, with 10 milligrammes of rasped copper, yielded, in the examination, only 8·6 milligrammes of copper,—loss, 1·4 milligrammes = 2·8 per cent.

Although many experiments have been performed to discover a shorter and better way for examination, all endeavors have afforded as yet no more satisfactory results than treatment with lead and boracic acid.

c. EXAMINATION OF ALLOYS OF COPPER AND ANTIMONY.

Among this order are found, particularly, the stibiferous alloys resulting from the examination of Grey Copper and copper ores containing no lead, and those obtained from rich copper ores, by the addition of oxide of antimony.

The separation of these metals is easily, and without loss, effected upon charcoal in the oxidating flame. The sample is placed in a cavity prepared on charcoal, and fused by a good oxidating flame, at the greatest possible distance, taking care to direct the flame aside, from time to time, in order to allow more free access of atmospheric air. The antimony volatilizes, the copper remaining pure. If, owing to the high per centage of antimony, the oxidation is continued for a long time, the depth of the cavity increases, so that the flame touches the alloy with difficulty; when this occurs, the process ought to be interrupted, and then continued on a new piece of charcoal.

The purity of the copper is recognized by its ductility, by its greenish-blue color in the fused, and its red copper color in the solid state. If the metallic globule does not possess these properties, it must be submitted to a new oxidation.

This sort of copper is generally argentiferous; it ought therefore to be examined for silver. The per centage of this metal is then deducted from the weight of the crude globule.

d. EXAMINATION OF ALLOYS OF COPPER AND TIN.

The compound of copper and tin, obtained by examinations of one decigramme of pure sulphide of tin, bell, and gun metal, belongs to this class of alloys.

Boracic acid cannot be employed advantageously to separate tin from copper, not only as it is fused with difficulty, if not in combination with oxide of lead, but also, because it scarcely acts as a solvent upon the tin. Better results are obtained by the use of a flux which combines fusibility with the property of dissolving the oxide of tin. Its composition is as follows, *videlicet*:—

Soda,	100 parts,
Boracic Acid,	50 parts,
Silica,	30 parts.

If stanniferous copper is to be examined, about 60 milligrammes of this composition are fused to a ball on charcoal, and placed by the side of the alloy. Of the compound obtained by reduction from one decigramme of sulphide of tin, the whole quantity is taken, while of the bell and gun metal, the quantity ought not to be larger than 45 to 50 milligrammes.

The glass bead, together with the alloy, is then fused in the reducing flame, until a rotatory motion ensues; from this the reducing is changed to the oxidating flame, which is so directed upon the glass, that it is protected from the access of air. The metallic globule then begins to oxidize on its surface, and if the oxide of tin, as well as of iron, is present, the latter is dissolved by the glass.

During the oxidation of tin, the sample ought to be kept in such a position, that the alloy may always be in contact with the charcoal on one, and with the fused glass on the other side, to prevent an oxidation of the copper. As this glass readily dissolves the tin, the process ought to be continued until it is perfectly saturated with the metal. Saturation is recognized by the formation of cavities in the enamelled glass, near which small particles of reduced tin appear. The solid metal is then removed from the fused scorice, and heated with 60 milligrammes of the above mentioned glass, upon another piece of charcoal, without detaching the small adhering portions of scorice, until the color of fused copper appears; at this period, the glass is covered with a pretty strong reducing flame, until it affords the properties of pure copper. It is then removed from the scorice, to observe its physical properties, color, and ductility. If it presents the

characteristic signs of pure copper, it may be weighed ; if not, it ought to be treated again, with 20 to 30 milligrammes of the flux.

In an examination of this nature, care must be taken not to oxidize a portion of the copper, together with the tin ; if this occurs, the suboxide of copper dissolved in the glass will communicate to it a brownish-red tinge. Such scoræ must be treated for some minutes with the reducing flame ; the copper is thus de-oxidized, combining with the main globule. A reduction of oxide of tin does not so soon take place, if the glass is not over-saturated. In the separation of the last portion of the tin from the copper, a quantity of the latter always oxidizes ; which, however, if proper care be taken, does not exceed the average of 0.3 milligramme upon 25 milligrammes.

IV.—THE LEAD ASSAY.

This metal is found in four different states of combination, in Minerals, Ore, Furnace, and Artificial Products,—

- a. As a metallic alloy ;
- b. As a sulphide ;
- c. Oxidized with mineral acids ;
- d. As a pure oxide, or only in combination with organic acids.

These four states of combination must be well considered in quantitative examinations with the Blowpipe, as it is sometimes necessary either to change certain substances in antecedent labors, or to separate wholly some constituents, before it is possible to render the lead pure. In substances containing the lead in the state of sulphide, its per centage may be ascertained in two different ways, *videlicet*—Substances containing other volatile ingredients besides sulphur, as arsenic, antimony, *et cetera*, are, as much as possible, purified from these bodies by calcination. The roasted sample is then mixed with soda and borax, placed between pulverized charcoal in two clay basins, one of which, serving as the smelting vessel, is covered with a paste of charcoal, next

submitted to the reducing flame, in order to de-oxidize the lead, and to scorify the other substances present in the assay. Here the reduced lead is scattered through the scoriae in granules of different sizes. The second treatment, employed only lately, is more simple, shorter, and likewise leads to exact results. Substances containing sulphides and arsenides, without antimony, are fused, without previous roasting, in a crucible, together with soda, borax, tartaric acid, and metallic iron; here the lead unites to one ball, the earthy and non-reducible oxides undergoing scorification.

The description of the first treatment would have been omitted, had it not been for its usefulness in cases where the presence of copper has to be determined.

With regard to the quantitative examination for lead, the minerals, ores, furnace and manufactured products may be divided into,—

- a. Such as contain the lead in a state of a sulphide;
- b. Such as contain it oxidized, and in combination with mineral acids;
- c. Oxides of lead, either pure or combined with organic acids;
- d. Metallic compounds.

a. **EXAMINATION FOR LEAD, IN MINERALS, ORES, AND PRODUCTS OF SMELTING WORKS, CONTAINING THIS METAL COMBINED WITH SULPHUR.**

First Method.

The substances belonging to this class, which can be examined quantitatively for lead by this method, are, among minerals—Lead Glance or Galena, Jamesonite, Bournonite, Zinkenite, *et cetera*; among ores dressed on the great scale—Galena, and all lead ores containing sulphides and arsenides of other metals; and among products of smelting works particularly—*bleistein, plumbiferous kupferstein, plumbiferous flue rakings, et cetera.*

A decigramme of these substances, reduced to a finely divided powder, should be weighed; and then freed from volatile ingre-

dients, by roasting on a pipe-clay capsule. As, however, the roasting of lead ores is similar to that of copper with charcoal powder, it will be omitted—except where a difference exists,—in order to avoid, as much as possible, unnecessary repetitions.

As soon as the roasting is ended, *id est*, when all the charcoal is burned off at a low red heat, and no more vapors of volatile ingredients are observed, the roasting capsule should be removed from the charcoal, the ore triturated in a mortar, and again mixed with twice its weight of charcoal powder. This mixture is to be spread on the pipe-clay capsule, and roasted a second time. As soon as the fire reaches the mixture, any escape of volatile ingredients will be readily ascertained by the smell. If this be the case, the charcoal should be allowed to burn off at a moderate red heat, the ore then triturated in a mortar, and roasted a third time with charcoal. It seldom happens that it is necessary to make three roastings; it cannot, however, be avoided, if the lead to be examined contains sulphides and arsenides, which are with difficulty decomposed. If no odor is observed during the ignition of the charcoal, in the second roasting, or only a slight odor of sulphurous acid, the assay may be considered to be fully roasted, as soon as the charcoal has been slowly consumed.

The roasting of pure lead glances, or rather such as contain neither sulphide of arsenic nor blende, proceeds much quicker than that of ores containing such ingredients. The cohesion of the particles of the ore is, however, less to be feared with the latter than with the former. Perfectly pure glances cannot be roasted *per se* with charcoal without *sintering*, and, therefore, something which hinders this from taking place must be added. This end is best effected, when 75 milligrammes of pure lead glance, containing about 80 per cent. of lead, are mixed with 25 milligrammes of pure pulverized pyrites, and this mixture then subjected to a second roasting with charcoal. As 100 milligrammes of galena, mixed in the above proportion with iron pyrites, would be too much, not only for the operation of roasting, but also for the subsequent reduction of the lead, this proportion may be changed according to the state of purity of the galena, so that the quantity of both substances together does not exceed

100 milligrammes. It is always very convenient, in calculating the per centage, that the lead resulting from the galena should be less than 100 milligrammes.

The characters of a perfectly roasted lead assay are as follows :—

1. The ignited assay, after the termination of the roasting, should give off no odor of sulphurous acid or arsenic.

2. It must exhibit a dull earthy appearance, without any shining particles of undecomposed sulphide of lead.

3. It would also be desirable that the assay be reduced to a loose friable state.

It is necessary to endeavor in some measure to recognize, from the appearance of the ores before roasting, as well as their color after undergoing this operation, not only the predominating admixed substances of the lead ores, but also to arrive at a conclusion as to the per centage of lead, as both have an important influence on the *dressing* required for the reduction of this metal. This is easily done, as pure galena, when roasted, has a greyish or yellowish-white color; the blendiferous galena, a greyish brown; and the pyritical, or ferruginous, a more or less red.

The dressing of the roasted lead ores, for the reduction of their oxide of lead, is very simple, depending principally on the other metallic oxides to be vitrified, and on the earthy constituents. The flux should consist of—

Soda, 100 milligrammes, and
Borax glass, 25 to 50 milligrammes.

The soda and the charcoal which surround the dressing during the fusion, serve as reducing agents for the oxide of lead. If other metallic oxides are contained in the assay, such as are reducible in this way give reguli, and those which are not are converted into protoxides.

The quantity of borax to be employed, is regulated by the quality of the ore. If the galena to be operated upon be in a *pure* state, it may be treated, after the roasting, with soda alone; or, if the slag formed during the fusion of the assay is one which the charcoal will not absorb, the mineral may then be treated with about 25 milligrammes of borax. The quantity of

borax increases in proportion with the poverty of the ore, or with the richness of the extraneous ingredients, as this flux is best adapted for dissolving most of the earthy constituents and irreducible metallic oxides. Thus, for example, in a mixture of galena, pyrites, arsenical pyrites, and blende, containing probably only from 3 to 5 per cent of lead, the latter cannot be perfectly separated during the fusion, when too small a proportion of borax has been employed, as, during the reduction of the lead, the slag formed is not sufficiently fluid to allow the finely divided metallic lead to separate and collect in large globules; a quantity of borax of at least 50 milligrammes should therefore be used.

The weighed soda and borax glass should be triturated with the roasted ores in an agate mortar, the powder shaken in the mixing capsule, and then introduced into a soda paper cornet, taking care to remove from the former, with a camel hair pencil, the adhering particles, in order that nothing may be lost. The cornet must be so closed, that the folded empty part,—which, in a packed dressed assay of silver or copper, is pressed up partly from both sides,—is in this case folded down on the full part, in order that it may have the form, as near as possible, of a hemisphere.

The assay thus prepared is then laid on a pipe-clay capsule, lined with charcoal, which should be already prepared, or formed immediately after the roasting, in order that it may dry during the weighing of the dressing. The assay should then be covered with just so much fine charcoal powder, that when a similar pipe-clay capsule is placed over it as a cover, the whole space between both capsules may be filled up.

If the charcoal has not been very much burned off by the roasting, it may still be employed for the fusion; but if, on the other hand, it has been so much burned that the necessary degree of heat cannot be attained, the part used should be removed, the capsule again placed in the charcoal holder, being arranged in the manner already given for the roasting of Copper Assays, and the platinum wire and foil attached to it.

The two capsules are then to be held in the forceps, and so placed on the charcoal, while the upper capsule is lightly pressed

with the two forefingers of one hand, that not only the rim of the under capsule rests on the edges of the cavity, but, at the same time, comes horizontally on the platinum wire, as is shown in *c*, Fig 27, page 27. If the capsule does not lie horizontally, the side which is lowest must be lifted up into the required position with the forceps, the upper capsule, at the same time, being kept in its position by the pressure of the finger. When the capsules are arranged in their proper position, the orifice in the charcoal holder must be closed, and the whole covered with a primitive piece of charcoal, having on its inner side a cavity *o*, three-eighths of an inch deep, formed with the largest of the charcoal borers, and a cylindrical opening *p*, three-tenths of an inch wide, which fits on to the charcoal holder, and which is kept in its place by the projecting sides of the latter.

When every thing is thus arranged, a strong oxidating flame should be directed through the circular opening *a* of the charcoal holder, which is to be held at a distance of from one and a half to two inches from the Blowpipe orifice. A stream of strongly heated air will by this means be forced into the empty part of the charcoal, which soon raises the inner sides, and also both capsules, to red heat. The temperature increases so rapidly, that a flame soon issues from the orifice *q*, in the primitive charcoal cover. When a Blowpipe nozzle with too small an orifice is not employed, it may be considered certain that the most difficultly fusible lead assay will be melted by an uninterrupted blast of five minutes, unless the charcoal be too hard, and thus prevent the necessary temperature from being readily obtained.

At the end of the proper time, the blast should be interrupted, the piece of charcoal used as a cover lifted off, and the platinum wire, with both capsules, removed from the charcoal with the workable lead tongs,—the wire should be caught firmly at *n*,—and placed on the anvil to cool. As soon as the assay has so far cooled that the capsules can be touched with the finger, the upper capsule is to be taken off, and the charcoal powder used as a protection, and also as a reducing agent, removed, and the fused assay examined. The full success of the operation may be judged of from the presence of the following characteristics :—

1. No sublimate of oxide of lead should appear near the orifice on the upper side of the charcoal, covering the assay. If this be the case, the heat has been too strong, and a portion of the lead has been volatilized.

2. The fused assay should be a perfect globular button, with a smooth surface.

3. The button must separate, on the slightest touch, from the almost uninjured charcoal powder with which the capsule is lined.

If the assay agrees with these characters, the button should be rolled in paper, placed on the anvil, struck lightly with the hammer, and again examined.

4. Although the slags of poor lead ores have a glassy fracture, such a fracture cannot be expected in the case of rich ores, because soda, with a slight addition of borax, does not yield vitreous slags with the small earthy or metallic constituents, particularly when silica is not present.

If the reduction has properly succeeded, the lead,—which very seldom occurs as a single globule, but generally as a number of large and small grains, disseminated through the slag,—should be separated from the latter in the following way :—The assay is to be again rolled in paper, and broken to a powder on the anvil. The paper is then to be carefully unfolded, the powdered assay shaken into a porcelain capsule, the larger grains of lead, free from slag, picked out, and the remainder of the lead separated from the slag by levigation, in the manner described for Copper Assays. If the large lead grains previously selected are not perfectly free from slag, they must be broken still smaller on the anvil, purified with water, and dried with the other lead grains, in a capsule.

In this way the whole of the lead is perfectly separated, but it still remains to be ascertained whether the lead, thus obtained, is pure from extraneous metals and other substances. This may be determined in the following manner :—

1. If the roasting has been carefully conducted, the lead must be free from sulphur and arsenic ; if not, a *bleistein* or *speiss* would be formed, which compounds would be reduced to powder

during the trituration of the slag, and may be distinguished from the lead after the levigation, with a magnifying glass, but cannot be mechanically separated from the latter without loss. When this occurs, the assay cannot be looked upon as successful, and the operation should therefore be repeated.

2. If the ore under examination was a pure galena, the resulting lead can contain no other metal but silver, as galenas in general, particularly those occurring on the sides of veins or lodes, are argentiferous.

3. If, on the other hand, it was an impure galena, with which copper pyrites and sulphide of antimony may occur in the assay powder, or a product of smelting operations composed of such ingredients, a portion of copper,—owing to the easy reducibility of oxide of copper,—and antimony—which does not volatilize during the roasting, but remains behind as antimonious acid—may be also present, for which the lead obtained must be examined. The presence of the latter metal, is, however, readily discovered in the resulting lead, by its being not only harder, but more brittle, and less malleable, than pure lead.

As, however, galena generally contains so little,—in case no rich silver ores are mixed with the dressed ores,—at most not more than from half to one per cent., and as copper and antimony rarely occur, with the exception of a few minerals, in dressed lead ores, a further separation of these metals from the resulting lead is only necessary, where it would be desirable to estimate, at the same time, the value of silver and copper.

When it is required to estimate accurately the value of the silver, copper, and antimony, contained in such plumbiferous ores, the resulting lead should be weighed on the balance, and examined for the previously mentioned metals in the following manner :—

1. For silver, it is only necessary to cupel on levigated bone ashes, in the manner described at pages 309, 310; the lead having been previously fused on charcoal, in the reducing flame, with borax glass.

2. Copper is found, when the lead is fused on charcoal, and treated with boracic acid, as given under that metal.

3. No accurate method of separating antimony in the dry way is known. Whether such an alloy contains antimony, may, however, be readily discovered, when treated on charcoal, in the reducing flame, by which the antimony is volatilized, and the charcoal coated with a white oxide.

The quantitative determination of the antimony present, which seldom amounts to more than one per cent., owing to the greater part being driven off during the roasting of the ore, can only be accomplished by the moist way ; but, as this would be too tedious, it needs not to be further mentioned.

After the examination of such alloys for silver or copper, the weight of the metals separated in the operation must be subtracted from the original weight of the compound, and the remainder considered as the true value of the lead contained in the assays under investigation.

Second Method.

The substances which can be analyzed quantitatively for lead by this method, are,—among minerals and ores dressed on the large scale, Galena, and all those lead ores which are mixed with sulphides and arsenides ; and among products of smelting works, *bleistein*, particularly, and plumbiferous *furnace rakings*, and also impure *roh* and *blei* slags.

The necessary assay powder should first be prepared from those substances, of which a quantity equal to one decigramme ought to be weighed. The quantity thus weighed should be shaken into a pipe-clay crucible, made as described at page 18, and well burned ; a piece of iron wire, about 50 milligrammes in weight, and of the thickness of a moderately strong knitting-needle, must then be laid on it. In order to prevent the cupel from falling, it may be placed in the small mould. Then weigh, of—

Soda, 150 milligrammes ;

Borax glass, 30 milligrammes ;

Tartaric acid, 20 milligrammes ;

triturate these ingredients in an agate mortar, and put the mix-

ture into the crucible containing the substance. The whole should then be covered with 50 milligrammes of dry chloride of sodium.

The iron serves to separate the sulphur and arsenic ; the soda and borax glass, for the production of the necessary slags, and solution of the other sulphides which may happen to be mixed with the sulphide of lead, and also of the earthy constituents ; the tartaric acid, for preventing the intumescence of the dressing in the crucible, as also, by its carbon, to keep the slags,—which, after the decomposition of sulphide of lead, generally contain sulphide of sodium,—from acting as a solvent on the metallic lead ; and, lastly, the chloride of sodium, for a cover, in order that the lead globules, separately set free, may the more readily combine, for which purpose it is admirably suited, owing to its great fluidity in the state of fusion, and its property of not combining with slags.

The ingredients employed for *the dressing*, can also be mixed with the substance to be examined ; but it has, however, been found that a larger number of globules of lead come to the surface of the slag in this case, and that it requires a longer time to combine them to one globule, than when the substance is placed with the iron *per se* in the bottom of the crucible.

The crucible, with its dressing, is then placed on a piece of charcoal, held on the charcoal holder, arranged in the same manner as for the roasting of a copper or lead ore, the platinum foil, however, not being used in this case, so that it rests quite free on the wire in the cavity ; *id est*, does not touch the charcoal, the rim of the crucible being on a level with the surface of the latter. The orifice of the charcoal holder is then closed, and the crucible covered with a piece of charcoal, arranged exactly in the same manner as in the fusion of a lead assay between two pipe-clay capsules.

When every thing has been thus arranged, a strong oxidating flame is deflected on the round orifice in the charcoal holder, at a distance of about two inches from the lamp, so that an intensely heated air passes into the hollow in the charcoal, by which the inner side and the crucible are raised to ignition, and the dressing

fused. If the charcoal is not too hard, the heat diffuses itself rapidly, and the assay is perfectly reduced in a blast of four, or at most five minutes' duration. As soon as the blast is discontinued, the upper charcoal or cover should be lifted, and the charcoal holder tapped with the broad side of the forceps, in order that the small globules of lead, that may happen to be disseminated through the slag,—which, however, rarely occurs,—may sink to the bottom, and combine with the main globule. The crucible is then to be removed from the charcoal, and placed for a few seconds in the small cupel mould to cool. As soon as it is cold, it must be broken with the hammer, on the anvil, and the lead and accompanying iron separated from the slag. The lead button is then caught with the forceps, placed on the anvil, so that the adhering iron may be upwards, and the latter,—which is sometimes, when the ore under examination is very pyritical, covered with sulphate of iron,—separated from the lead, by a few slight percussions. The lead button, freed from iron, may be purified from any adhering alkaline slag, by flattening on the anvil, between moistened filtering paper; and then weighed.

If it be suspected that the substance under examination contains silver or copper, the former may be separated by cupellation on bone ashes, and the latter by treatment with boracic acid; and the weight of the metals thus separated subtracted from the original weight of the assay.

The value of the silver thus obtained, when the substance is a pure galena, is equal to the value deduced from a special assay for silver, but it is too small when the substance is an argentiferous pyrites, or contains any other argentiferous sulphide. The value of copper is generally too small; the cause of this is easily understood, and therefore requires no further explanation.

If the substance under examination,—as, for example, iron pyrites, arsenical pyrites, blende, or *roh* and *blei* slags, with finely disseminated particles of *rohstein*, *et cetera*,—contain only from one to ten per cent. of lead, it is always difficult to separate the lead from the iron, so that its weight can be ascertained with accuracy. In such cases, a fine, accurately weighed silver button, of about 50 to 80 milligrammes, should be added

to the dressing ; and instead of 20 milligrammes of tartaric acid, 50 milligrammes should be added to the slag, which may probably still contain oxide of lead. The reduced lead combines with the silver, and may be separated from the iron, and its value ascertained by again weighing the silver button, and subtracting the weight of the original silver button,—the remainder being the value of the lead.

When such substances are rich both in silver and copper, the assay succeeds by the first method.

If an ore prepared on the great scale, consisting principally of galena, should contain a plumbiferous ore, in which the lead is contained in an oxidized state, 100 milligrammes of tartaric acid should be added instead of 20, in order that a sufficient quantity of carbon may be present to reduce the oxide of lead.

b. ESTIMATION OF THE LEAD IN MINERALS, ORES, AND ARTIFICIAL PRODUCTS, CONTAINING THIS METAL IN THE STATE OF OXIDE, COMBINED WITH MINERAL ACIDS.

To this division belong the *green*, *brown*, *yellow*, and *red* varieties of earthy carbonate of lead, sulphate of lead, and artificial chromate of lead, or the commercial chrome-yellow.

All these compounds are characterized by being either not at all, or only partly decomposed by roasting, as the stability of many of the acids is too great, and their affinity for the oxide of lead too strong.

If lead salts, free from other salts, particularly metallic sulphates, and also earthy ingredients, are to be assayed for lead, they may be considered as nearly pure roasted galena. An assay powder being first prepared, a decigramme is to be dressed with—

Soda, 100 milligrammes ;

Borax Glass, 25 milligrammes ;

and subjected to treatment similar to what has been already

given for Lead Assays which require to be roasted previous to reduction.

If, on the other hand, the acidiferous lead ores contain other metallic salts, particularly sulphates, or if combinations of metallic sulphides and arsenides be mixed with them, a perfect roasting with charcoal is necessary.

A decigramme of the substance, so roasted, should be dressed with—

Soda, 100 milligrammes ;

Borax Glass, 40 to 50 milligrammes.

The further treatment of such an assay remains, however, the same.

If acidiferous lead ores are mixed with other metallic salts, the bases of which are reduced with the lead, the latter must be examined for silver, copper, and antimony, in the manner above described.

In the reduction of oxide of lead from lead salts, the soda, with the surrounding charcoal, serves as a reducing agent for the oxide of lead and the acids. Thus, for example, arsenic acid is reduced to metallic arsenic, which volatilizes ; sulphuric acid, to sulphur, which combines with the sodium of a portion of the soda, forming sulphide of sodium ; chromic acid, to oxide of chromium, which permeates the slag. Borax, however, serves only, as with pure galena, to prevent the absorption of the soda by the charcoal support ; as it fuses with soda, and probably with reduced portions of the ores, to a bead, while the metallic lead, in various sized globules, collects on its surface.

In the reduction of the oxide of lead, contained in a roasted assay composed of lead salts, contaminated with other substances, the soda plays the same part as in the preceding case, in addition to its solvent action, which it probably exerts on traces of silica ; but an excess of borax, however, principally serves as a solvent for the non-reducible metallic oxides.

c. METHOD OF ESTIMATING THE VALUE OF LEAD, IN PRODUCTS OF
SMELTING WORKS,

*And other Artificial Substances containing Lead in a pure oxidized state,
or only combined with Vegetable Acids.*

To this class belong *litharge*, *abstrich*, *cupel grounds*, pure *roh* and *blei* slags, all kinds of plumbiferous glass, white lead, sugar of lead, *et cetera*.

The bodies of this class do not require to be roasted ; but in dressing them for the reduction of their oxide of lead, it should be ascertained whether the body under examination is a simple oxide of lead, or a combination of oxide with other substances.

As the preceding substances may be at once subjected to the reduction process, they should be dressed,—the pure oxide of lead, or such oxides of lead as are merely combined with organic acids, with

Soda, 100 milligrammes ;

Borax Glass, 25 milligrammes ;

and those in which difficultly fusible ingredients predominate, as for example, *cupel grounds*, *roh* and *blei* slags, with a quantity of borax amounting to about 50 per cent.

If such products contain, besides lead, other easily reducible metals, the resulting lead button, after being weighed, must be examined for such metals in the manner already given.

d. METHOD OF ESTIMATING THE VALUE OF LEAD, IN MINERALS
CONTAINING THIS METAL ALLOYED WITH OTHERS.

Black Tellurium Yellow Tellurium, Selenide of Lead, Selenide of Lead and Copper, and Selenide of Lead and Mercury, come under this class.

Of these minerals, the German author had only an opportunity of examining selenide of lead quantitatively for its metal

before the Blowpipe, and the method by which its value in this mineral was found, can only be given.

The assay succeeds most readily, and most accurately, when 100 milligrammes of the finely powdered mineral are introduced into a pipe clay crucible, with a small piece of iron of 30 to 50 milligrammes in weight, covered with a mixture of 150 milligrammes of soda, 30 milligrammes of borax glass, and 20 milligrammes of tartaric acid ; on which mixture, about 50 milligrammes of dried chloride of sodium should be shaken, and the fusion then proceeded with in the manner described at pages 370, 371. If the selenide of lead contains an admixture of sulphide of lead, it will be decomposed ; the selenium, however, combines with the radical of the soda, forming selenide of sodium, from which a small portion of the selenium volatilizes, and the lead, combined in one globule, deposits on the iron, at the bottom of the crucible. When the fusion is finished, and the fused assay has cooled, the crucible is broken, and the reduced lead freed from the iron and slag. The malleability of the lead button should be tested, and its weight ascertained.

When required, the value of the silver present may be ascertained by cupelling the button on bone ashes, and weighing the resulting silver button, when sufficiently large ; if too small for the balance, it should be measured on the scale.

V.—DETERMINATION OF TIN.

The usual method of determining tin by the dry way, which has been for so long a time employed, and is so still in most assay laboratories, is far from being accurate. There are a great many different methods of determining quantitatively the value of tin in an ore, by the dry way ; but if it be a ferruginous tin ore, by one method pure tin will be obtained, but generally in too small a quantity, while by another, it appears in excess, but never pure. It must be here observed, that in practice, where it is certain that too much, and therefore impure tin, is obtained, the value is

taken at a few per cents. less than that obtained by experiment. The cause of this is, probably, that with a sufficient quantity of carbon, the reduced tin acts during the fusion as a reducing agent on the sesquioxide of iron, by which a portion of the latter is not only reduced to protoxide, but even to the metallic state, which latter combines with the reduced tin, affording a hard tin button, which, when weighed, gives too high a result.

The German author, after convincing himself, by a number of tin assays made according to different methods, of the above source of error, concluded, that these discrepancies could not be avoided in all the control assays made according to the usual methods, and for this reason he introduced a new one.

For this purpose, various quantities of pure peroxide of tin were mixed with a number of pulverized minerals,—as Iron Pyrites, Sulphide of Arsenic, Blende, *et cetera*, which generally occur along with tin ores ;—and the value of tin which each should afford, calculated ; they were then submitted to the action of the Blowpipe, with the view to determine this value by a number of experiments. The assays made with these mixtures fully convinced him that the same difficulties are to be encountered with the Blowpipe, as with the usual tin assays by the dry way, made on a large scale. From one to two, and once even four per cent. too much,—that is, of ferriferous tin, were obtained. A number of other experiments, in which all possible means to slag perfectly as protoxide in the dry way by fusion the peroxide, which would be formed during the roasting, were employed but in vain ; in no case was the resulting tin free from iron. At length, after trying every other way, the iron was separated in an oxidized state from the oxide of tin by a simple yet accurate means in the moist way, namely, by hydrochloric acid, and the oxide of tin left behind with the earthy ingredients was then submitted to reduction.

By the employment of this method, a pure malleable tin was always obtained, whose weight agreed well with that found by calculation. Mixtures of the pure tinstones with other ores, containing either copper or iron, yield exactly the same result.

It may be objected to the employment of hydrochloric acid for

the separation of oxide of tin from oxide of iron, that it does not belong to Blowpipe analysis ; but if the difficulties which are to be encountered in the dry way with the Blowpipe, in order to purify peroxide of tin from the oxides of iron and copper, be compared with the easy and accurate method by the moist way, there is no doubt the latter will be chosen, as the best which can be employed in the quantitative examination of ferruginous and cupriferous minerals and dressed ores for tin.

In respect to the quantitative examination for tin ;—minerals, ores, and artificial products, in which this metal forms an essential ingredient, may be classed as follows :—

- a.* Such as contain the tin combined with sulphur ;
- b.* Those containing the tin in an oxidized state ; and,
- c.* Those in which metallic tin is alloyed with other metals.

a. DETERMINATION OF TIN IN MINERALS, ORES, AND ARTIFICIAL PRODUCTS, IN WHICH THE TIN IS COMBINED WITH SULPHUR.

Besides Tin Pyrites, Tin Schlichs, dressed on the large scale, may be placed in this division ; although they contain tin in the state of oxide, they very often, notwithstanding the roasting, exhibit traces of metallic sulphides and arsenical compounds.

Of the artificial products which belong to this class, Mosaic Gold—sulphur combined with excess of tin, will only be mentioned.

To determine the tin in any of the substances belonging to this class, an assay powder should be prepared according to the method given at page 298,—100 milligrammes of which are to be weighed and roasted, in order to free it from volatile ingredients. But as the roasting of tin assays is exactly similar to those of copper with charcoal dust, any further notice of it here is unnecessary.

If the volatile bodies contained in a substance to be roasted be confined to sulphur, or to sulphur with a trace of arsenic, or only a few per cents. of arsenic, which last is sometimes the case with

tin schlichs, prepared on the great scale, the roasting is very soon finished ; but if a large quantity of arsenic is present, the roasting with charcoal is longer, and must be continued until not the slightest odor of arsenic can be observed, when a fresh portion of charcoal is added, and the mass kept in a state of ignition for some time. Tin schlichs, dressed on the great scale, if already roasted, require to be again roasted only once ; the other substances, on the other hand, in which the tin is combined with sulphur, or mixed with metallic sulphides or arsenical compounds, require to be roasted with charcoal two or three times.

By this process of roasting, when carefully performed, sulphur, arsenic, the greater part of the antimony, and a small portion of the zinc, will be volatilized from a stanniferous substance, mixed with, probably, Iron Pyrites, Arsennikkies, Copper Pyrites, Speissglanz, Blende, Wolfram, *et cetera* ; while the other metals, tin—when it is not present in an oxidized state—copper, iron, manganese, and the remainder of the zinc, will be oxidized. Those metals, exclusive of arsenic, which are capable of acidification, and which are difficultly or not at all volatilized during the roasting,—as, for example, a small portion of the antimony, molybdenum, tungsten, titanium, *et cetera*,—remain behind as acids.

The characters which distinguish a well roasted tin ore, are exactly similar to those of a well roasted lead one. Thus, the roasted ore should yield no odor in contact with charcoal in a state of ignition ; and no shining particles of metallic sulphides or arsenical compounds should appear, when the substance, after the charcoal is perfectly burned away, is rubbed in a mortar ; it should also be perfectly friable on the roasting capsule.

If a well roasted tin ore, consisting, for example, of the oxides of tin, iron, manganese, and copper, be immediately subjected to reduction, a brittle, grey, and too heavy tin button is obtained, even with the best fluxes and reducing agents, owing to the oxide of copper being at the same time reduced, affording a brittle metallic compound ; and, secondly, as above mentioned, to a portion of the oxide of iron being reduced to the metallic state, and which likewise combines with the tin.

As the disadvantages just mentioned cannot, however, be

guarded against in the dry way, it is better to remove the oxides of iron, manganese, and copper, from the roasted tin ore, with hydrochloric acid. This end may be attained in the following manner :—

The well roasted ore is to be introduced into a small porcelain crucible—see Fig. 41—spread out thinly on its bottom, and hydrochloric acid poured on it, the quantity depending on the substances to be dissolved ; the vessel is then placed on the triangle, at about two inches and a half over the flame of the spirit-lamp, the wicks of which should be pressed so far into the wick-holder, that only a very small flame is afforded, just sufficient to warm the crucible strongly ; in order to prevent the fumes from escaping into the room, it should be covered.

The acid should then be observed, whether it is colored from dissolved metallic oxides, and whether small bubbles rise here and there from the substance, which show that it has commenced to boil. The digestion should be conducted in this manner, uninterruptedly, for four or five minutes, taking care that the acid does not boil too strongly, as in that case particles of matter may be lost. The vapors which arise during the solution are generally condensed on the cover and fall back again into the solution.

After the expiration of at most five minutes, in which time all the oxides of iron, manganese, copper, and zinc, and also the antimonious acid, will be dissolved out, the triangle and crucible should be removed from the lamp, and the whole allowed to cool.

The cover should then be taken off, and the adhering drops of acid removed from it with bibulous paper. The clear yellow or green supernatant solution, resting on the undissolved powder, which consists of only oxide of tin, or tinstone,—sometimes, however, contaminated with earthy matter, or with tungstic and titanitic acids,—is then to be drawn off with a pipette, and replaced with from three to four times as much water. The water should always be poured down the side of the vessel, so that the substance on the bottom be not disturbed, and the light particles suspended in the fluid, as otherwise the deposition of these particles again would require some time.

In order to remove the whole of the colored solution, the vessel should be again heated over the lamp, and nearly the whole of the water drawn off with the pipette, by slightly inclining the crucible to one side.

If very minute particles of the powder, which are generally earthy ones, should float about on the surface of the water, and which sink with difficulty to the bottom, the fine orifice of the pipette, in removing the water, should be kept about a quarter of an inch under the surface, in order that nothing may be lost.

The rest of the water is separated from the substance by placing the capsule on the triangle over the spirit-lamp, until the powder is perfectly dry.

The whole operation,—by which, with necessary caution, no loss of tin need be apprehended,—does not, including the time employed in solution, require at most more than a quarter of an hour.

That the protoxides of iron and manganese, chemically combined with the tinstone, which scarcely amount together to from two to three per cent., even in an exceedingly dark colored tinstone, cannot be removed in this way, is easily seen; but as this protoxide of manganese, and the greater part of the protoxide of iron, are dissolved by the slag in the reduction, only an unappreciable trace of iron in the metallic state combines with the reduced tin.

The next operation is the reduction of the oxide of tin, resulting from the roasting, or in an unchanged state of tinstone, freed from admixed metallic oxides by hydrochloric acid. This is effected by the aid of the proper fluxes and reducing agents, in a space surrounded by charcoal, just in a similar manner to the reduction of oxide of lead, in roasted lead ores. The dressing for this purpose is very simple, consisting of—

Soda, 100 milligrammes;

Borax Glass, 30 milligrammes.

This dressing is to be mixed with the ore in an agate mortar; the mixture is then to be introduced into a soda paper cornet, like roasted lead ores, dressed with soda and borax—see page 365—and fused between two pipe-clay capsules. Tin assays,

however, require to be fused for eight minutes. When the assay has cooled, a bead is found in the bottom of the capsule, consisting of slag, in which the reduced tin is enclosed, sometimes as one, but oftener disseminated through the mass in various sized globules. These globules, like those of lead and copper, are freed from slag by levigation with water, and then dried.

The purity of the resulting tin may be judged of from its action on the magnet, its color, and its malleability.

If the roasting, and the separation of the oxides of iron and copper, and the antimonious acid be carefully conducted, a pure tin of the proper weight may always be obtained by employing the necessary degree of heat in the reduction. If the ore has not been properly treated with hydrochloric acid, a brittle tin is obtained after the reduction, when copper and antimony are present; and when a little iron is present, the tin obtained, although malleable, acts on the magnet when in a finely divided state under water, the weight of which is naturally too high. If the ore under examination contains tungstic or titanitic acids, they cannot be separated by means of hydrochloric acid, as already mentioned; but as they combine with soda in the reduction of the peroxide of tin, they produce no injurious effect on the result.

If the metal obtained has the properties of pure tin, it can be weighed; if not, a fresh assay must be more carefully performed.

Tinstone occurs in *interlaced masses, flat or dilated veins* and gangues, or vein stones, with Granite, Gneiss, Micaceous Schist, Litharge, Talk Clay, Calcareous Spar, and many other minerals and rocks, as also with Iron and Arsenical Pyrites, Speiss Glanz, Blende, Wolfram, Molybdic Ochre, Iron Ochre, Magnetic Iron Ore, but, in a very large number of these minerals, only in so finely disseminated a state that the presence of tin in them can scarcely be suspected, and cannot be ascertained with accuracy, even by a trial with the Blowpipe, owing to the minute quantity present; but by careful levigation and decantation of such an ore, reduced to a fine powder, and weighed, the earthy particles may be removed, and a schlich obtained in which the tin ore is concentrated, and from which, when perfectly dried and weighed, at least two quantitative tin assays should be made according to

the manner described ; the resulting tin is then weighed, tested as to its purity, and the value for the undressed ore calculated.

Thus, for example, if 5000 milligrammes of such a fine pulverized ore yield by levigation with water, and subsequent decantation, a quantity weighing 700 milligrammes when dried, from which, when thoroughly mixed and triturated in an agate mortar, two tin assays are prepared ; now, if both these assays give 1·5 per cent. of tin, there would be contained in the 700 milligrammes of schlich—

$$100 : 700 :: 1\cdot5 : x ;$$

or, $1 : 7 :: 1\cdot5 : x = 7 \times 1\cdot5 = 10\cdot5$ milligrammes of tin. If the levigation has been carefully performed, these 10·5 milligrammes represent very nearly the whole value of the tin contained in the above 5000 milligrammes of raw ore, which gives the quantity in 100 at—

$$5000 : 100 :: 10\cdot5 : x ;$$

$$\text{or, } 50 : 1 :: 10\cdot5 : x = \frac{10\cdot5}{50} = 0\cdot21 \text{ per cent. of tin.}$$

b. DETERMINATION OF TIN IN MINERALS AND PRODUCTS CONTAINING THIS METAL IN AN OXIDIZED STATE.

To this class belong, of minerals, pure Tinstone ; and of artificial products, *tin ashes*,—the dross left on the floor of the reverberatory furnace in the refining of tin—enamel, *et cetera*.

Such substances do not require roasting, nor, if oxides of iron, copper, or antimony be not accidentally present, the treatment with hydrochloric acid, previous to the reduction of the oxide of tin. It is merely necessary to weigh 100 milligrammes of the perfectly dried and finely pulverized tinstone, or of such artificial products as are not combined with silicic acid ; to dress them with—

Soda, 100 milligrammes ;

Borax Glass 30 milligrammes ;

and subject them to reduction in the same manner as the substances belonging to the preceding class.

To determine the value of tin in enamel, where the oxide is in

combination with silicic acid, 100 milligrammes of the substance should be dressed with—

Soda, 150 milligrammes ;

Borax Glass 30 milligrammes ;

by which the silicic acid combines with soda, and the oxide of tin is reduced. But as oxide of lead is often present in enamel, and is also very easily reduced, the resulting tin will not be pure, owing to its combining with a portion of lead. Such a compound cannot be separated into its elements in the dry way, but with nitric acid the lead can be dissolved out, leaving the tin as oxide undissolved. It is then only necessary to edulcorate this oxide, dry it, ignite it strongly in a platinum spoon, and calculate the value of metal from the weight of the ignited oxide,—100 parts of oxide of tin containing 78·62 parts of metallic tin.

c. DETERMINATION OF TIN IN STANNIFEROUS ALLOYS.

To this class belong bell and gun metal, and all combinations of tin with lead, bismuth, zinc, and antimony. But as the quantitative determination of tin in the latter compounds in the dry way, with the Blowpipe, is very uncertain, while in the moist way, on the other hand, it may be easily and accurately determined, the application of the Blowpipe is therefore confined to bell and gun metal, which are compounds of tin with copper.

The method of separating tin from copper has been already fully described, in speaking of the Copper Assay at page 360. In this method, the copper is the principal object of investigation, no reference being made to the tin. But if it is required to find the value of tin at the same time, the glass which contains the whole of the tin as oxide should not be lost, because the metal may be again reduced from it.

When desirable to determine the tin before the Blowpipe, in a combination of tin and copper, the tin must be oxidized by the method previously described, and in this state separated, together with the flux, consisting of soda, borax, and silica. This glass should then be pulverized, mixed with about 50 milligrammes of

soda, this mixture introduced into a soda paper cornet, and fused, surrounded with charcoal, between two pipe-clay capsules, like an ordinary tin assay. But the tin is found, after the fusion, contaminated with a trace of copper, owing to an exceedingly small quantity of the latter combining with the glass in this operation.

The resulting tin is now to be weighed, and the centesimal value found by calculation, as only 50 milligrammes of such metallic compounds are generally employed in an assay.

APPENDIX.

SYSTEMATIC ARRANGEMENT

OF THE

OXIDIZED MINERALS,

ACCORDING TO THEIR BEHAVIOR BEFORE THE BLOWPIPE.

.

WHEN the nature of a mineral is to be ascertained by its behavior before the Blowpipe, the external characters not being sufficiently distinct for that purpose, it is important, especially for young mineralogists, that some arrangement should be contrived, by which the number of species, with which the mineral under examination is to be compared, should be diminished, so as to be as small as possible. Such a systematic arrangement, which, although deficient in many respects, will not be without use, is now given. Three properties of minerals; namely, fusibility, intumescence, and behavior with soda, have been especially considered. The principal divisions depend on the former two properties, the subdivisions on the latter.

1. *Infusible Minerals.*

Give with soda a bead.	Give with a small quantity of soda a bead, but form a slag with more.	Give a slag only with soda.
Quartz	Phenakite	Oxides of iron
Agalmatolite	Picrosmine	Oxides of manganese
Hisingerite	Olivine	Oxide of tin—is reduced
Sideroschistolite	Cerite	Hydrate of alumina
Diopase	Kyanite	Hydrate of magnesia
Fire clay	Talc	Spinel
Leucite	Gadolinite	Gahnite
Pyrophyllite	Lithia tourmaline	Wörthite
Wolkonskoite		Precious galmei
Rutile		Pitchblende
		Zircon
		Thorite
		Andalusite
		Staurotide
		Gehlenite
		Chlorite spar
		Allophane
		Cymophane
		Polymignite
		Aeschynite
		Oerstedine
		Titanate of iron
		Yttrio-tantalite
		Tantalite
		Chrome ochre
		Uwarowite
		Chromic iron
		Carbonates of earths and metals
		Basic phosphate of yttria
		Basic phosphate of alumina
		Basic phosphate of lime
		Sulphate of iron
		Sulphate of alumina
		Aluminite
		Alum stone
		Fluoride of cerium
		Yttrio-cerite
		Topaz

2. *Minerals which can be fused to beads.*

Fuse with soda to a bead.	Give with a small quantity of soda a bead, with more a slag.	Give a slag only with soda.
Zeolites	Okenite	Brevicite
Spodumene	Pectolite	Amphodelite
Labradorite	Red silicate of manganese	Chlorite
Scapolite	Black silicate of manganese	Fahlunite
Sodalite, from Greenland	Idocrase	Pyrope
Elaolite	Manganesian garnet	Soapstone
Mica, from primitive Limestone	Orthite	Red dichroite
Black talc	Pyrorthite	Pyrargillite
Achmite	Amblygonite	Potassa tourmaline, black
Crocidolite	Sordawallite	Wolfram
Lievrte	Sodalite,	Pharmacolite
Cronstedtite	Fluor Spar	Scorodite
Garnet		Cube ore
Cerine		Tetraphylline
Helvine	Give with soda a reguline metal.	Hetepozite
Gadolinite, from Karsarvet		Uranite
Boracic acid	Tungstate of lead	Phosphate of iron
Tincal	Molybdate of lead	Sulphate of strontia
Boracite	Vanadate of lead	Sulphate of magnesia
Hydroboracite	Chromate of lead	Polyhallite
Datholite	Vauquelinite	Haumyne
Botryolite	Cobalt bloom	
Axinite	Nickel bloom	
Lapis lazuli	Phosphate of copper	
Eudialyte	Sulphate of lead	
Pyrosmalite	Chloride of lead	
Cryolite	Chloride of silver	

3. *Minerals which can be fused only on their edges.*

Give with soda a liquid bead.	Give with a small quantity of soda a liquid bead, but with more a slag.	Give a slag only with soda.
Soapstone	Table spar	Stilpnosiderite
Felspar	Diallage	Plumbobresinite
Albite	Hypersthene	Serpentine
Petalite	Epidote	Silicate of manganese, from Piedmont
Nepheline	Zoisite	Mica, from granite
Anorthite		Pimelite
Emerald		Pinite
Euclase		Blue dichroite
Calaite		Carpholite
Sodalite, from Vesuvius		Sphene
		Pyrochlore
		Wolfram
		Soda tourmaline, green
		Lazulite
		Heavy spar
		Gypsum

4. *Minerals which intumesce.*

Fuse to a bead.	Become alaggy on the edges.	Infusible.
Zeolites	Epidote	Gadolinite, some varieties
Onkosine	Zoisite	Lithia tourmaline
Boracite	Green tourmaline	Aeschynite
Hydroboracite	Lazulite	Pyrophyllite
Datholite	Plumbo resinite	Alum
Botryolite	Granite mica	Sulphate of alumina
Axinite	Hornblende	
Black silicate of manganese	Euclase	
Lithia spodumene	Sphene	
Scapolite		
Eleolite		
Idocrase		
Cerine		
Orthite		

BEHAVIOR OF URINARY CALCULI BEFORE THE BLOWPIPE.

It is highly important to medical men to be able to determine the nature of the urinary calculi of patients who apply to them for relief in such cases. Their composition is more readily ascertained than is generally supposed, since the Blowpipe gives very simple and sufficiently accurate results, and the use of this instrument does not require a greater degree of chemical knowledge than every physician ought to possess.

1. URIC ACID CALCULI.

Per se on charcoal or on platinum foil they become carbonized, fume, and emit an organic smell ; in the exterior flame they gradually diminish in size, and towards the end of the operation they burn with increased light. When the blast is discontinued, they still continue to glow, and at last leave a very slight trace of a white ash, which is strongly alkaline.

Since other combustible substances may easily be mistaken for uric acid, the operation must be performed in the moist way, as follows :—a small quantity of the calculus, say one-tenth of a grain, must be placed on a small strip of glass or platinum foil, moistened with a few drops of nitric acid, and heated over the flame of the spirit-lamp. The uric acid dissolves with effervescence, and the mass must then be carefully dried so as not to become burnt, and then heated for an instant, when the mass ac-

quires a fine red color. If the quantity of uric acid in the assay is trifling, the latter often becomes black, without acquiring a tinge of red. The experiment must then be repeated, and when the solution in nitric acid is dry, it must be laid aside, and allowed to cool perfectly; the glass must next be inverted over a little caustic ammonia, which is then to be heated; as soon as the vapor of the ammonia reaches the dried substance, it becomes beautifully red. The same thing may be observed, though the color is less striking, if the dried substance be softened with diluted ammonia.

It often happens that calculi contain a mixture of uric acid with earthy phosphates. They then carbonize and consume, leaving a considerable residuum, which is not alkaline or soluble in water. Treated with nitric acid, and afterwards with ammonia, they give the fine red color characteristic of uric acid. The remaining ash is either phosphate of lime or phosphate of magnesia, or a mixture of the two.

2. URATE OF SODA CALCULI.

This substance, which rarely occurs in urinary calculi, is often contained in the hard excrescences which form around the joints of gouty patients.

Alone on charcoal they blacken, emit a burnt organic odor, and leave a grey, strongly alkaline substance, which can be fused to a glass with silica. If the calculus contains earthy salts, as is usually the case, the glass becomes white, or greyish, and opaque.

3. URATE OF AMMONIA CALCULI.

They behave before the Blowpipe like the uric acid calculi. Treated with a drop of caustic potassa, they give, when slightly warmed, a strong smell of ammonia. Care must be taken not to confound this with the light ammoniacal odor which almost all

organic substances develop when treated with an alkali. These calculi often contain a quantity of urate of soda.

4. PHOSPHATE OF LIME CALCULI.

Alone on charcoal they blacken, emit the odor of burning organic matter, and finally burn white. They do not fuse. Their behavior resembles in general that of phosphate of lime.

They are proved not to consist of silica, by swelling up with soda and not fusing to a glass, as well as by giving a regulus of phosphide of iron, when dissolved in boracic acid and fused with iron.

5. AMMONIACAL PHOSPHATE OF MAGNESIA CALCULI.

Heated alone on platinum foil they smell strongly of salts of ammonia, blacken, intumesce, and if the heat be increased, burn greyish-white. They fuse readily to an enamel-like bead of the same color.

They give a transparent glass with borax and microcosmic salt, which, with a large quantity of the assay, becomes opaline on cooling.

With soda they fuse to a white, swollen slag, which becomes infusible on the addition of more of the flux.

With boracic acid and iron they give readily a regulus of phosphide of iron.

With nitrate of cobalt they give a dark red glass.

6. CALCULI COMPOSED OF THE PHOSPHATES OF LIME AND MAGNESIA.—*Calculi fusibiles.*

These are recognized by the slight ammoniacal odor they emit, arising principally from the destruction of organic matter in the calculus. They fuse far more readily than any others to an enamel-white bead, which does not become red with the oxide of cobalt, but brownish-black.

7. CALCULI COMPOSED OF OXALATE OF LIME.

Calculi moriformes.

Alone they smell of urine when first heated. The more strongly crystalline varieties become duller and lighter colored. After being slightly heated, the residuum effervesces with a few drops of nitric acid, and after being strongly heated on charcoal caustic lime remains; this has an alkaline reaction on reddened litmus paper, and generally crumbles to powder when moistened. This is not the case, however, if the calculus contains at the same time phosphate of lime.

8. CALCULI CONTAINING SILICA.

They leave after ignition a greyish, infusible, and sometimes slaggy ash, which, when mixed with soda, slowly dissolves to a more or less transparent glass.

9. CYSTIC OXIDE CALCULI.

These behave, before the Blowpipe, very much like calculi composed of uric acid; they do not fuse, but take fire readily, and burn with a bluish-green flame, emitting a strongly acid smell of a peculiar character, somewhat resembling that of hydrocyanic acid. They leave an ash which is not alkaline, and which fuses in a strong heat to a greyish-white mass.

They are distinguished from uric acid calculi by the smell which they emit when burning, and by not giving a red color with nitric acid.

TABLE OF ATOMIC WEIGHTS.

NAME.	SYMBOL.	ATOMIC WEIGHT.
		Oxygen, 8.
Aluminum	Al	14
Ammopium. . . .	N H ⁴	18
Ammonia	N H ⁴ O	26
Antimony	Sb	129
teroxide of	Sb O ³	158
tersulphide of	Sb S ³	177
Arsenic	As	75
tersulphide of	As S ³	123
pentasulphide of	As S ⁵	155
Arsenic acid	As O ⁵	115
Arsenious acid	As O ³	99
Barium	Ba	68.5
chloride of	Ba Cl	104
Baryta	Ba O	76.5
carbonate of	Ba O, C O ³	98.5
sulphate of	Ba O, S O ³	116.5
Bismuth	Bi	213
teroxide of	Bi O ³	287
Borium	B	11
Bromine	Br	80
Cadmium	Cd	56
Calcium	Ca	20
chloride of	Ca Cl	55.5
Carbon	C	6
Carbonic acid	C O ²	22

NAME.	SYMBOL.	ATOMIC WEIGHT.
		Oxygen, 8.
Carbonic oxide. . . .	C O	14
Cerium	Ce	46
Chlorine	Cl	35.5
Chromium	Cr	28
Cobalt	Co	29.5
Copper	Cu	32
protoxide of	Cu O	40
suboxide of	Cu ² O	72
Fluorine	Fl	19
Glucinum	Gl	18
Gold	Au	199
Hydrogen	H	1
Iodine	I	126
Iridium	Ir	99
Iron	Fe	27
sesquioxide of	Fe ³ O ³	78
protoxide of	Fe O	35
Lead	Pb	104
carbonate of	Pb O, C O ³	134
chloride of	Pb Cl	139.5
phosphate of	2 Pb O, P O ⁵	368
protoxide of	Pb O	112
sulphate of	Pb O, S O ³	152
Lime	Ca O	28
carbonate of	Ca O, C O ³	50
sulphate of	Ca O, S O ³	68
Lithium	Li	6.5
Magnesia	Mg O	21
sulphate of	Mg O, S O ³	61
Magnesium	Mg	13
Manganese	Mn	28
protoxide of	Mn O	36

NAME.	SYMBOL.	ATOMIC WEIGHT.
		Oxygen, 8.
Manganese, sesquioxide of	$\text{Mn}^2 \text{O}^3$	80
Mercury	Hg	100
subchloride of	$\text{Hg}^2 \text{Cl}$	235.5
chloride of	Hg Cl	135.5
oxide of	Hg O	108
suboxide of	$\text{Hg}^2 \text{O}$	208
Molybdenum	Mo	48
Nickel	Ni	29.5
Nitrogen	N	14
Osmium	Os	100
Oxygen	O	8
Palladium	Pd	53
Phosphoric acid	P O^3	72
Phosphorous acid	P O^3	56
Phosphorus	P	32
Platinum	Pt	99
ammonio-chloride of	$\left\{ \begin{array}{l} (\text{Pt Cl}^2 + \\ \text{N H}^+ \text{Cl}) \end{array} \right\}$	223.5
potassio-chloride of	$\left\{ \begin{array}{l} (\text{Pt Cl}^2 + \\ \text{K Cl}) \end{array} \right\}$	244.5
Potassa	K O	47
carbonate of	K O, C O ²	69
sulphate of	K O, S O ²	87
Potassium	K	39
chloride of	K Cl	74.5
Rhodium	R	52
Selenium	Se	40
Silicium	Si	15
Silver	Ag	108
chloride of	Ag Cl	143.5
oxide of	Ag O	116
Soda	Na O	31
carbonate of	Na O, C O ²	53

NAME.	SYMBOL.	ATOMIC WEIGHT.
		Oxygen, 8.
Soda, sulphate of	Na O, S O ^a	71
Sodium	Na	23
chloride of	Na Cl	58.5
Strontia	Sr O	52
carbonate of	Sr O, C O ^a	74
sulphate of	Sr O, S O	92
Strontium	Sr	44
chloride of	Sr Cl	79.5
Sulphur	S	16
Sulphuric acid	S O ^a	40
Sulphurous acid	S O ^a	32
Tantalum	Ta	185
Tellurium	Te	64
Thorium	Th	60
Tin	Sn	59
binoxide of	Sn O ^a	75
protoxide of	Sn O	67
Titanium	Ti	24.5
Tungsten	W	95
Uranium	U	217
Vanadium	V	69
Water	H O	9
Yttrium	Y	32
Zinc	Zn	32
oxide of	Zn O	40
Zirconium	Zr	22.5

INDEX TO THE MINERALS

ENUMERATED IN THE SECOND SECTION.

- АсНИМІТЪ, 105, 175, 224
Actynolite, 115
Aeschynite, 140, 154, 219, 224
Agalmatolite, 103
Alalite, 115
Albite, 105, 181
Allanite, 116, 132, 154
Allophane, 181, 192
Aluminate of Magnesia, 125
Aluminite, 130
Alum, 102
Alum-alate, 132
Analcime, 105, 181
Amblygonite, 105, 130
Ammonalun, 107, 130
Amphibole, 115, 126
Anatase, 224
Anauxite, 132
Andesine, 105
Andalusite, 132
Anhydrite, 113
Anorthite, 115, 116, 125, 131
Anthracite, 249
Anthraconite, 113
Antigonite, 126
Anthosiderite, 176
Antimonbloom, 210
Antimonial Grey Copper, 191, 209
Antimonial Lead Glance, 209
Antimonial Nickel, 169, 231
Antimonial Ochre, 209
Antimonial Silver, 193, 200
Antimonocker, 103
Antimonasilber Blende, 198
Atrimolite, 103
Apatite, 114
Apoklas, 114
Apophyllite, 102, 114
Aphrodite, 126
Aquerite, 200
Argent Rouge Arsenie, 231
Argentiferous Copper Glance, 190, 199
Argillaceous Ironstone, 174
Arkansite, 224
Arragonite, 110, 118
Arseniate of Iron, 174, 231
Arseniate of Lead, 231
Arsenical Antimonial Silver, 231
Arsenical Antimony, 209, 231
Arsenical Iron, 174
Arsenical Nickel, 168, 231
Arsenical Pyrites, 231
Arsenical Silver, 198, 209
Arsenicbluthe, 231
Arsenik Speisglanz, 231
Arsenioderite, 176
Arsensilber Blende, 198
Arvedsonite, 105
Asbestiform Actynolite, 115
Asbestos, 115
Asbestos Tremolite, 115
Atacamite, 191
Aurichalcite, 192
Automalite, 130, 163, 174
Axinite, 116, 132
Axotomous Antimony Glance, 182
Asurite, 191
Asure Stone, 116
Babingtonite, 116
Batrachite, 126
Barowite, 116

- Barytocalcite, 108, 113
 Baryta-harmatome, 108
 Basic Chloride of Lead, 183
 Besumontite, 116
 Berg Crystal, 251
 Berg Milch, 113
 Berthierite, 209
 Beryl, 181, 138
 Bismuth Blende, 231
 Bismuth Cobalt Ore, 186
 Bismuth Glance, 186
 Bismuth Ochre, 186
 Bismuthic Silver, 186, 199
 Black Earthy Cobalt, 160, 166
 Black Garnet, 128
 Black Lead Ore, 183
 Black Tellurium, 182, 205, 229
 Black Wad, 160
 Black Yttrio-Tantalite, 114, 140, 215
 Bleierde, 183
 Bleigummi, 132
 Bleiglanz, 164
 Bleihorners, 184
 Bleiglatte, 184
 Bleischimmer, 184
 Bloodstone, 174
 Blue Carbonate of Copper, 191
 Bodenmais Tantalite, 161
 Bodenite, 154
 Bog Iron Ore, 174
 Bolus, 132
 Baltonite, 126
 Boracite, 125
 Borax, 105
 Botryolite, 114
 Boulangerite, 184
 Bournonite, 182, 191, 209
 Braunitz, 108, 161
 Brevicite, 105
 Broddbo-Tantalite, 161
 Brookite, 224
 Brochantite, 192
 Brewsterite, 108, 110
 Bright White Cobalt, 166, 231
 Brittle Silver Glance, 198
 Brittle Sulphide of Silver, 191, 198
 Brown Arsenical Lead Ore, 183
 Brown Earthy Cobalt, 166
 Brown Coal, 249
 Brown Iron Ore, 174
 Brown Menaccanite, 114
 Brown Spar, 125
 Brown Yttrio-Tantalite, 215
 Brucite, 125
 Buchsinite, 132
 Bunt-kupfererz, 190
 Byssolite, 115
 Bytownite, 105
 Calamine, 164
 Calcareous Heavy Spar, 108, 113
 Calcareous Spar, 113
 Carbonate of Bismuth, 186
 Carbonate of Cerium, 154
 Celestine, 110
 Cerine, 116, 182, 154
 Cerite, 164
 Chabasite, 103, 105, 130
 Chalk, 113
 Chalcedony, 252
 Chalkolite, 188, 192
 Chamoisite, 175
 Chloride of Lead, 183
 Chloride of Mercury, 201
 Chloride of Silver, 199
 Chloride of Ammonium, 107
 Chlorite, 126, 181
 Chloropal, 161, 175
 Chlorophaeit, 175
 Chromate of Iron, 174, 227
 Chromate of Lead, 183, 227
 Chrome Iron Ore, 227
 Chrome Ochre, 227
 Chrom-mica, 103
 Chrysoberyl, 181, 138, 227
 Chrysolite, 169, 175
 Chrysocola, 192
 Cimolite, 132
 Cinnabar, 201
 Clay, 132
 Clay Slate, 132
 Cobalt Bloom, 166, 231
 Cobalt Kies, 166
 Columbite, 219
 Common Pyrites, 174
 Comptonite, 105
 Condrodite, 125, 126
 Condurrite, 191, 231
 Copper Blende, 174, 191
 Copper Glance, 199
 Copper Pyrites, 174, 190, 199
 Cordierite, 126
 Cornelian, 251
 Corundum, 130
 Cotunnite, 188
 Couzzeranite, 103
 Crichtonite, 174, 224
 Cronstedtite, 176
 Cryolite, 130
 Cryptolite, 164
 Cuban, 192
 Cube Ore, 175, 231
 Cupreous Bismuth, 186, 191
 Cupreous Manganese, 191
 Cupriferos Sulphide of Bismuth, 186
 Cuproplumbite, 192
 Cyanite, 130
 Cymophane, 181, 138, 224
 Dark Ruby Silver, 198, 209

Dark Yttrio-Tantalite, 114, 140
 Datolite, 114
 Delvauxite, 176
 Dermatine, 126
 Desmine, 114
 Diamond, 249
 Diamant Spar, 130
 Diaspore, 130
 Dichroite, 126, 131
 Dihydrate, 192
 Diopside, 115
 Dioptase, 192
 Diploite, 103
 Dodecahedral Iron Ore, 160, 174
 Dolomite, 113, 125
 Dreeelite, 108

 Earthy Carbonate of Lead, 183
 Edingtonite, 116
 Egeran, 115
 Ehlite, 192
 Eisenapatite, 161, 176
 Eisengranate, 176
 Eisensand, 224
 Eisensinter, 231
 Ekaolite, 103, 131
 Emerald, 131, 138, 237
 Epidote, 115, 130
 Epistilbite, 105
 Erbia, 141
 Erinite, 191, 231
 Esmarkite, 126
 Euclase, 131, 138
 Euchroite, 191, 231
 Eudyalite, 149
 Eugen Glance, 198
 Eukairite, 190, 198, 237
 Eukolite, 219
 Euxenite, 140, 154, 189, 219, 224

 Fahlers, 192, 210, 231
 Fahlunite, 105
 Fayalite, 176
 Federerz, 184
 Felspar, 102, 131
 Ferriferous Malacolite, 115
 Ferriferous Pyroxenes, 115
 Fergusonite, 140, 149, 154, 189, 219
 Finbo-Tantalite, 161
 Fibroferrite, 176
 Flint, 251
 Fluoride of Cerium, 154
 Fluoride of Yttrium, 154
 Fluor-spar, 113
 Fowlerite, 161
 Franklinite, 160, 164, 174
 Fuller's Earth, 132

 Gadolinite, 138, 140, 154, 176
 Gahnite, 130, 163

Galmei, 181
 Gansekothigerz, 191
 Gaylussite, 104
 Gehlenite, 115, 131
 Gelb-bleierz, 183, 213
 Geokronite, 184, 210
 Gibbsite, 130
 Gigantolite, 103
 Gilbertite, 132
 Glauberite, 105, 113
 Glauber Salt, 104
 Glaukolite, 105
 Glottalite, 116
 Gmelinite, 103, 105
 Gneiss, 132
 Grammatite, 115
 Garnet, 115, 131, 132, 161, 175
 Graphic Tellurium, 198, 204, 229
 Graphite, 249
 Graugiltigerz, 174, 199, 209
 Green Arsenical Lead Ore, 183
 Green Earth, 126, 131
 Greenockite, 181
 Green Uranite, 188
 Grey Antimony, 191, 209
 Grey Copper, 174, 191, 199, 209, 231
 Grey Oxide of Manganese, 160
 Grovoville, 161
 Grüneisenstein, 175
 Gymnite, 126
 Gypsum, 113

Haarkies, 169
 Halloysite, 132
 Hardingerite, 114, 174
 Harmotome, 115, 131
 Harringtonite, 105
 Hausmannite, 108, 161
 Heavy Spar, 108, 110
 Hedenbergite, 115
 Helvine, 138, 161
 Hemiprismatic Olive Malachite, 183
 Hemiprismatic Ruby Blende, 198
 Hepatic Cinnabar, 201
 Hetoposite, 161, 176
 Heulandite, 114
 Hisingerite, 175
 Holmite, 116
 Honeystone, 132
 Hornblende, 115
 Horn Quicksilver, 201
 Horn Silver, 199
 Horn Stone, 251
 Huraulite, 161, 176
 Huronite, 116
 Humboldtite, 103, 105, 176
 Hyacinth, 149
 Hydrophite, 217
 Hydrous Aluminate of Lead, 183

Idiocrase, 115, 131
 Ilmenite, 174, 224, 227
 Indianite, 105
 Iodic Mercury, 201
 Iodic Silver, 199
 Iolite, 126
 Iron Alum, 180
 Isarine, 174, 224

Jamesonite, 182, 209
 Jeffersonite, 164

Kakosene, 175
 Kalk-tuff, 113
 Kaolin, 132
 Karpholite, 131, 161
 Kieselkupfer, 192
 Kieselzinkerz, 164
 Küberckenite, 184, 210
 Kirwanite, 116
 Kimito-Tantalite, 161
 Klinoklas, 192
 Knebelite, 161, 176
 Kobellite, 184, 210
 Kollyrite, 132
 Krokydolite, 175
 Kryolite, 104, 130, 132
 Kupferblende, 209
 Kupferglanz, 190
 Kupferkies, 192
 Kupfermanganerz, 121
 Kupfernickel, 168, 231
 Kupferwismutherz, 192
 Kyanite, 224
 Kyrosite, 176

Labradorite, 105, 115, 131
 Lapis Lazuli, 116
 Lavendulan, 169
 Lazulite, 116, 130, 132
 Lazurstein, 116
 Laumonite, 115, 131
 Lead Glance, 182
 Lebererz, 201
 Lepidolite, 106
 Leucite, 102, 131
 Leuchtenbergite, 126
 Leucophane, 138
 Libethenite, 191
 Lichtes Rothgiltigerz, 198
 Lievrite, 115, 175
 Light Ruby Silver, 198, 209, 231
 Limestone, 113
 Liroconite, 191, 231
 Litharge, 171
 Lithion Spodumene, 106
 Loam, 132

Magnesia-idiocrase, 126
 Magnesia-pyroxene, 126

Magnetic Iron Pyrites, 173
 Magnesite, 125
 Maclureite, 125
 Melaccolite, 115
 Malachite, 191
 Malthacite, 132
 Manganblende, 160
 Mangan-glance, 161
 Manganite, 161
 Manganocalcite, 161
 Mansfield Copper Slate, 217
 Marl, 113, 132
 Mascagnin, 107
 Meerschau, 125
 Melan-glance, 209
 Melanochroite, 184, 227
 Menaccanite, 114, 174, 224
 Mesolite, 131
 Mesotype, 131
 Mica, 131, 224
 Mica Slate, 132
 Miloschine, 132, 227
 Molybdate of Lead, 183, 213
 Molybdic Acid, 203
 Monasite, 154
 Monazitoid, 154
 Monradite, 126
 Mosandrite, 154, 224
 Murchisonite, 103
 Mysorine, 192
 Myargyrite, 198, 209

Nadelers, 192
 Nakrite, 132
 Native Amalgam, 198, 201
 Native Antimony, 209
 Native Arsenic, 231
 Native Bismuth, 185
 Native Blue Vitriol, 191
 Native Boracic Acid, 250
 Native Copper, 190
 Native Gold, 198, 203, 204
 Native Green Vitriol, 174
 Native Iridium, 203
 Native Iron, 168, 173
 Native Mercury, 201
 Native Oxide of Copper, 191
 Native Peroxide of Manganese, 160
 Native Platinum, 203
 Native Saltpetre, 239
 Native Silver, 198
 Native Sulphate of Lead, 183
 Native Tellurium, 205, 229
 Natron, 104
 Nematite, 126
 Nepheline, 103, 105
 Nickel Glance, 169, 231
 Nickel Ochre, 166, 169, 231
 Nickeliferous Grey Antimony, 209
 Nickelspeiss Glanzers, 169, 231

- Nigrin, 174, 224
 Nitrate of Soda, 105, 240
 Noble Molybden Glance, 205
 Nontronite, 176
 Nosin, 116, 132
 Nussierite, 184
 Nuttallite, 103

 Octohedral Arseniate, 191
 Oerstedite, 149, 224
 Okenite, 116
 Oligoklas, 105
 Olivenite, 192
 Olivine, 125, 169, 175
 Orpiment, 231
 Orthite, 116, 132, 140, 154
 Osmium Iridium, 203
 Ottrelite, 132
 Oxide of Chrome, 227
 Oxide of Arsenic, 231
 Oxidulated Iron Ore, 174

 Parallolite, 125
 Parisite, 154
 Pea Iron Ore, 217
 Pechstein, 105
 Pechuran, 189
 Peganite, 132
 Pemelite, 156
 Pennine, 126
 Periklas, 125
 Perouskite, 224
 Petalite, 106, 131
 Phacolite, 103
 Pharmacolite, 114, 231
 Phenakite, 138
 Pholerite, 132
 Phosphate of Iron, 175
 Phosphate of Manganese and Iron, 160
 Phosphate of Yttria, 130
 Phosphoro-chalcite, 191
 Picramine, 107, 125
 Pimelite, 169
 Pinguite, 176
 Pinite, 103
 Piottine, 126
 Pissophane, 132
 Pistacit, 115, 131
 Pitchblende, 188, 217
 Pitchy Iron Ore, 175, 231
 Plagionite, 184, 210
 Pleonaste, 125
 Plinthite, 132
 Plombiferous Black Copper, 184
 Plumbo-cupriferous Sulphide of Bismuth, 183
 Plombgomme, 130, 133
 Polianite, 161
 Polybasite, 192, 198
 Polyargite, 103

 Polyhallite, 102, 113, 125
 Polykras, 140, 149, 154, 189, 219, 224
 Polymignite, 140, 149, 154, 224
 Porphyry, 132
 Potassa Alum, 130
 Potassa Harmatome, 115
 Potassa Tourmaline, 102
 Potstone, 126, 131
 Praseolite, 126
 Prehnite, 115, 131
 Prismatic Copper Mica, 191
 Prismatic Lead Spar, 183
 Prismatic Magnesian Mica, 126
 Prismatic Titanic Ore, 224
 Pseudo-Malachite, 191
 Psilomelan, 103, 161
 Purple Copper, 174, 190
 Pynknite, 132
 Pyramidal Tellurium Glance, 182
 Pyramidal Tin Ore, 207
 Pyrrargillite, 103
 Pyrope, 227
 Pyrochlore, 140, 149, 152, 154, 188, 219, 224
 Pyrolusite, 161
 Pyrorthite, 116, 132, 140, 154
 Pyrosklerite, 227
 Pyrosomalite, 161, 175
 Pyroxenes, 115

 Quartz, 251

 Ramlite, 132
 Ramphillite, 103
 Realgar, 231
 Red Antimonial Ore, 209
 Red Ironstone, 174
 Red Oxide of Copper, 191
 Red Oxide of Zinc, 164
 Red Siliciferous Oxide of Manganese, 161
 Red Silver, 198
 Red Vitriol, 166
 Retinalite, 105
 Rhodalite, 176
 Rhombohedral Ruby Blande, 209
 Rhombohedral Molybdena Glance, 213
 Rhomboidal Arseniate, 231
 Rock Milk, 113
 Rock Salt, 104, 107, 246
 Romeite, 210
 Rosite, 103
 Rothbleierz, 227
 Rothrinkerz, 164
 Rothkupfererz, 191
 Ruby, 130
 Rutile, 224
 Ryakolite, 103

- Sahlite, 115
 Saltpetre, 102
 Samarskite, 219
 Sandstone, 132
 Sapphire, 130
 Saussurite, 105
 Sassoline, 107
 Scapolite, 115, 131
 Scheelite, 183
 Scheelbleispath, 215
 Schieferspar, 113
 Schillerspar, 126, 227
 Schorl, 102
 Schriftern, 200
 Schwartz-kupfer, 169
 Schwarzbleierz, 183, 184
 Schwerstein, 114, 215
 Scolezite, 115, 131
 Selenblei, 237
 Selenite, 113
 Selenite of Cobalt and Lead, 166, 237
 Selenide of Copper, 190, 237
 Selenide of Copper and Lead, 182, 190
 Selenide of Lead, 182, 199, 237
 Selenide of Lead and Copper, 182, 192, 237
 Selenide of Lead and Mercury, 182, 237
 Selenide of Silver and Copper, 198
 Selenilber, 200
 Serpentine, 116, 125, 227
 Siberian Needle Ore, 169, 183, 186, 191, 229
 Sideroschisolite, 175
 Silberkupferglanz, 190
 Silicious Oxide of Zinc, 164, 181
 Sillimanite, 182
 Silver Glance, 198
 Skorodite, 174, 231
 Slate Spar, 113
 Soapstone, 126, 131
 Sodalite, 105, 131
 Soda Felspar, 105
 Soda Spodumene, 105
 Soda Tourmaline, 105
 Sordawallite, 126
 Spadaite, 126
 Spathose Iron, 175
 Specular Iron, 174
 Speckstein, 125
 Speisecobalt, 169, 231
 Sphene, 114, 224
 Spinel, 125, 130, 227
 Spinellane, 116
 Splendent Fibrous Blende, 181
 Spodumene, 105, 131
 Sproglaserz, 200
 Staurolite, 131
 Steatite, 126
 Sternbergite, 176, 200
 Stilbite, 114, 131
 Stilpnomelan, 176
 Stinkstone, 113
 Stone Coal, 249
 Striegisane, 132
 Stromnite, 110
 Strontianite, 110
 Sulphate of Magnesia, 125
 Sulphate of Potassa, 102
 Sulphate of Zinc, 164
 Sulphide of Arsenic, 231
 Sulphide of Mercury, 201
 Sulphide of Nickel, 169
 Sulphide of Silver, 198
 Sulphide of Silver and Arsenic, 198
 Sulphide of Silver and Copper, 198
 Swedish Lead Glance, 237
 Symplectite, 176
 Tabular Spar, 114
 Tachylite, 103, 107
 Tagalite, 192
 Talc, 126, 131
 Tamela-Tantalite, 161
 Tantalite, 161, 176, 215, 219
 Tarandite, 125
 Tellurblei, 200
 Telluric Bismuth, 186, 229, 237
 Tellurilber, 200
 Tennantite, 174, 190, 231
 Tephroite, 161
 Terbia, 141
 Tetartine, 105
 Tetrahedral Copper Glance, 209
 Tetraphylite, 161, 176
 Thomsonite, 105
 Thoneisenstein, 174
 Thonerde, 132
 Thorite, 152, 189
 Thrombolite, 192
 Thuringite, 176
 Tin Pyrites, 191, 207
 Tinstone, 207, 219
 Tin White Cobalt, 166
 Tincal, 105
 Titaneisen, 224
 Titanic Acid, 224
 Titaniferous Oxidulated Iron, 174
 Titanite, 114, 124
 Topaz, 132
 Tourmaline, 106, 132
 Tremolite, 115
 Triphylite, 161, 176
 Triplite, 160, 175
 Tritomite, 154
 Troostite, 161
 Trona, 104
 Tschewkinite, 154, 224
 Tuesite, 132
 Tufa, 113

Tungstate of Lead, 183, 215

Tungstate of Lime, 215

Tungstic Acid, 215

Tutty, 164, 184

Umber, 161

Uranbloom, 189

Uranite, 188

Uran Ochre, 188

Uranotantalum, 189

Uranpechers, 188

Uranvitriol, 189

Urao, 104

Urwarowite, 227

Vanqueline, 183, 192, 227

Vanadate of Lead, 183, 217

Vanadate of Lime, 217

Vanadiniferous Lead Spar, 183

Variscite, 182

Varvicite, 161

Vesuvian, 115

Villarsite, 126

Vitreous Copper, 190

Vitriol-oker, 175

Vivianite, 176

Volbartite, 192, 217

Volcanic Iron, 224

Voltsite, 164

Wad, 160

Wavellite, 180

Weissbleierz, 183

Weissgiltigerz, 174, 184, 191, 199, 209

Weissite, 103

Weissnickel, 168

Weissnickelkie, 231

Weiss-silvanerz, 198

Weisstallur, 200

Werkblei, 184

Wernerite, 115

White Antimony, 209

White Cobalt, 166

White Lead Ore, 183

Witherite, 107

Wohlerite, 105, 149, 219

Wolchonskite, 227

Wolfram, 160, 174, 215

Wollastonite, 114

Worthite, 132

Wurfelerz, 176

Xanthokon, 200

Xenolite, 132

Yellow Earthy Cobalt, 166

Yellow Menaccanite, 114

Yellow Tellurium, 182, 198, 205, 229

Yellow Yttrio-tantalite, 114, 140, 188

Yenite, 115

Yttria-phosphate, 140

Yttrocerite, 140, 154

Yttrotantalite, 114, 140, 219

Yttrotitanite, 141, 224

Zeagonite, 103

Zeylanite, 125, 130

Zinc Blende, 163, 181

Zinc Bloom, 164

Zinciferous Spinel, 164

Zinkenite, 183, 209

Zinnkie, 193

Zircon, 149

Zoisite, 115, 180

ERRATA.

- Page 52, bottom line, *for* emerald-green, *read* amethystine-violet.
,, 126, line 21, *for* Leuchtenbergerite, *read* Leuchtenbergite.
,, 131, ,, 4, *for* Anacime, *read* Analcime.
,, 176, ,, 8, *for* Humboldtite *read* Humboldtite.
,, 192, ,, 24, *for* Blestein, *read* Bleistein.
,, 192, ,, 28, *for* Kuperwismuthers, *read* Kupferwismuthers.
,, 225, ,, 3, *for* Titantiferous, *read* Titaniferous.
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A CLASSIFIED INDEX TO MR. CHURCHILL'S CATALOGUE.

ANATOMY.

	PAGE
Anatomical Remembrancer ..	3
Beale on Liver ..	5
Hassall's Micros. Anatomy ..	14
Holden's Human Osteology ..	15
Jones' and Sieveking's Pathological Anatomy ..	17
MacLise's Surgical Anatomy ..	19
Paget's Catalogue ..	21
Sibson's Medical Anatomy ..	25
Wheeler's Handbook ..	30
Wilson's Anatomy ..	30

CHEMISTRY.

Abel & Bloxam's Handbook ..	3
Bowman's Practical Chemistry ..	7
Do. Medical do. ..	7
Chalmers' Electro-Chemistry ..	8
Fownes' Manual of Chemistry ..	12
Do. Actonian-Prize ..	12
Do. Qualitative Analysis ..	12
Fredericus' Chemical Analysis ..	12
Galloway's First Step ..	12
Do. Analysis ..	12
Do. Diagrams ..	12
Griffiths' Four Seasons ..	13
Horaley's Chem. Philosophy ..	16
Jones.—Mulder on Wine ..	17
Odling's Practical Chemistry ..	21
Plattner on Blowpipe ..	22
Spear's Pathol. Chemistry ..	26

CLIMATE.

Francis on Change of Climate ..	12
Hall on Torquay ..	14
Haviland on Climate ..	14
Lee on Climate ..	18
Martin on the Undercliff ..	19
Martin (J. R.) on Tropical ..	20

DEFORMITIES, &c.

Biggs on Deformities ..	6
Bishop on Deformities ..	6
Do. Articulate Sounds ..	6
Brodhurst on Spine ..	7
Do. on Clubfoot ..	7
Hare on Spine ..	14
Hugman on Hip Joint ..	16
Inman on Spine ..	16
Tamplin on Spine ..	26

DENTISTRY.

Blundell's Painless Extraction ..	7
Clark's Odontalgist ..	9
Gray on the Teeth ..	13
Odontological Soc. Transactions ..	21
Tomes' Dental Surgery ..	28

DISEASES of the URINARY and GENERATIVE ORGANS, and SYPHILIS.

	PAGE
Acton on Reproductive Organs ..	3
Coots on Syphilis ..	9
Coulson on Bladder ..	10
Do. on Lithotomy ..	10
Egan on Syphilis ..	11
Gant on Bladder ..	13
Judd on Syphilis ..	17
Mander on Chancre ..	20
Milton on Gonorrhoea ..	20
Parker on Syphilis ..	21
Todd on Urinary Organs ..	28
Wilson on Syphilis ..	31

DISEASES OF WOMEN AND CHILDREN.

Barker on Children ..	4
Bennet on Uterus ..	5
Do. on Uterine Pathology ..	5
Bird on Children ..	6
Brown on Women ..	7
Do. on Scarlatina ..	7
Eyre's Practical Remarks ..	11
Hood on Crowing ..	16
Lee's Ovarian & Uterine Diseases ..	18
Lee on Diseases of Uterus ..	18
Do. on Speculum ..	18
Robertson on Women ..	23
Rowe on Females ..	24
Smith on Leucorrhoea ..	25
Tilt on Diseases of Women ..	27
Do. on Change of Life ..	27
Underwood on Children ..	29
West on Women ..	30

HYGIENE.

Armstrong on Naval Hygiene ..	4
Beale's Laws of Health ..	5
Do. Health and Disease ..	5
Bennet on Nutrition ..	5
Blundell's Medicina Mechanica ..	6
Carter on Training ..	8
Cornaro on Long Life ..	10
Hartwig on Sea Bathing ..	14
Do. Physical Education ..	14
Hufeland's Art ..	16
Lee's Watering Places of England ..	18
Do. do. Germany, France, and Switzerland ..	18
Lee's Rhenish Watering Places ..	18
Parkin on Disease ..	21
Pickford on Hygiene ..	22
Robertson on Diet ..	24
Rumsey's State Medicine ..	24
Van Oven's Decline of Life ..	29
Wilson on Healthy Skin ..	31
Do. on Mineral Waters ..	31

MATERIA MEDICA and PHARMACY.

	PAGE
Bateman's Magnaoplia ..	4
Beasley's Formulary ..	5
Do. Receipt Book ..	5
Do. Book of Prescriptions ..	5
Lane's Materia Medica ..	18
Pereira's Selecta e Prescriptis ..	22
Pharmacopoeia Londinensis ..	22
Prescriber's Pharmacopoeia ..	22
Royle's Materia Medica ..	24
Spurgin's Materia Medica ..	26
Steggall's Materia Medica ..	26
Do. First Lines for Chemists ..	26
Stowe's Toxicological Chart ..	26
Taylor on Poisons ..	27
Wittstein's Pharmacy ..	31

MEDICINE.

Acland on Cholera ..	3
Adams on Rheumatic Gout ..	3
Addison on Supra-Renal Capsules ..	3
Addison on Cells ..	3
Alexander on Rheumatism ..	3
Baly and Gull on Cholera ..	4
Barclay on Diagnosis ..	4
Barlow's Practice of Medicine ..	4
Basham on Dropsy ..	4
Beale on Urine ..	5
Billing's First Principles ..	6
Bird's Urinary Deposits ..	6
Bird on Charcoal ..	6
Brinton on Stomach ..	7
Do. on Ulcer of do. ..	7
Budd on the Liver ..	7
Do. on Stomach ..	7
Campbell on Diabetes ..	8
Chambers on Digestion ..	8
Davey's Ganglionic ..	10
Eyre on Stomach ..	11
Fuller on Rheumatism ..	12
Gairdner on Gout ..	12
Granville on Sudden Death ..	13
Gully's Simple Treatment ..	13
Habershon on Stomach ..	13
Hall on Apnea ..	13
Hall's Observations ..	13
Harrison on Lead in Water ..	14
Hassall on Urine ..	14
Headland on Medicines ..	15
Hooper's Medical Dictionary ..	16
Hooper's Physician's Vade-Mecum ..	13
Jones' Animal Chemistry ..	17
Lugol on Scrofula ..	19
Peacock on Influenza ..	21
Pynn on Yellow Fever ..	23
Roberts on Falsy ..	23
Robertson on Gout ..	24
Savory's Compendium ..	24
Sample on Cough ..	24
Shaw's Remembrancer ..	25
Smee on Debility ..	25
Steggall's Medical Manual ..	26

CLASSIFIED INDEX.

MEDICINE—continued.

	PAGE
Steggall's Gregory's Conspectus	26
Do. Celcus	26
Thomas' Practice of Physic	27
Thudichum on Urine	28
Wegg's Observations	29
Wells on Gout	29
What to Observe	19
Whitehead on Transmission	30
Williams' Principles	30
Wright on Headaches	31

MICROSCOPE.

Beale on Microscope in Medicine	5
Do. How to Work	5
Carpenter on Microscope	8
Schacht on do.	24

MISCELLANEOUS.

Action on Prostitution	3
Atkinson's Bibliography	4
Bascome on Epidemics	4
Bryce on Sebastopol	7
Cooley's Cyclopaedia	9
Davy's (Sir H.) Remains	11
Forbes' Nature and Art in Disease	12
Guy's Hospital Reports	13
Haycock's Veterinary	15
Lane's Hydropathy	18
Marcel on Food	19
Massy on Recruits	20
Part's Case Book	21
Pettigrew on Superstitions	22

NERVOUS DISEASES AND INDIGESTION.

Carter on Hysteria	8
Child on Indigestion	8
Downing on Neuralgia	11
Hunt on Heartburn	16
Lobb on Nervous Affections	19
Radcliffe on Epilepsy	23
Reynolds on the Brain	23
Rowe on Nervous Diseases	24
Sieeking on Epilepsy	25
Todd on Nervous System	28
Turnbull on Stomach	28

OBSTETRICS.

Barnes on Placenta Praevia	4
Davis on Parturition	11
Lee's Clinical Midwifery	18
Pretty's Aids during Labour	22
Ramsbotham's Obstetrics	23
Do. Midwifery	23
Sinclair & Johnston's Midwifery	25
Smellie's Obstetric Plates	25
Smith's Manual of Obstetrics	25
Swayne's Aphorisms	26
Walier's Midwifery	29

OPHTHALMOLOGY.

	PAGE
Cooper on Injuries of Eye	9
Do. on Near Sight	9
Dalrymple on Eye	10
Dixon on the Eye	11
Hogg on Ophthalmoscope	15
Holthouse on Strabismus	15
Do. on Impaired Vision	15
Jacob on Eye-ball	16
Jago on Ocular Spectres	16
Jones' Ophthalmic Medicine	17
Do. Defects of Sight	17
Do. Eye and Ear	17
Nunneley on the Organs of Vision	21
Walton on Ophthalmic	29

PHYSIOLOGY.

Carpenter's Human	8
Do. Comparative	8
Do. Manual	8
Cottle's Human	10
Heale on Vital Causes	15
Hilton on the Cranium	15
Richardson on Coagulation	23

PSYCHOLOGY.

Arlidge on Lunacy	3
Bucknill and Tuke's Psychological Medicine	8
Burgess on Madness	7
Burnett on Insanity	9
Conolly on Asylums	9
Davey on Nature of Insanity	10
Dann's Physiological Psychology	11
Hood on Criminal Lunatics	16
Jacobi on Hospitals, by Tuke	28
Millingen on Treatment of Insane	20
Monro on Insanity	20
Do. Private Asylums	20
Noble on Psychology	20
Do. on Mind	20
Williams (J.) on Insanity	30
Williams (J. H.) Unsoundness of Mind	30
Winslow's Lettsomian	31

PULMONARY and CHEST DISEASES, &c.

Addison on Healthy and Diseased Structure	3
Billing on Lungs and Heart	6
Blakiston on the Chest	6
Bright on the Chest	7
Cotton on Consumption	10
Do. on Stethoscope	10
Davies on Lungs and Heart	10
Dobell on the Chest	11
Fenwick on Consumption	11
Laennec on Auscultation	17
Markham on Heart	19
Richardson on Consumption	23
Skoda on Auscultation	19
Thompson on Consumption	27
Turnbull on Consumption	28
Weber on Auscultation	29

SCIENCE.

	PAGE
Bird's Natural Philosophy	6
Burnett's Philosophy of Spirits	8
Craig on Electric Tension	10
Garner's Eutherpela	13
Hardwich's Photography	14
Hinds' Harmonies	15
Jones on Vision	17
Do. on Body, Sense, and Mind	17
Mayne's Lexicon	20
Nourse's Students' Tables	21
Price's Photographic Manipulation	22
Rainey on Shells	23
Reymond's Animal Electricity	23
Taylor's Medical Jurisprudence	27
Vestiges of Creation	28
Sequel to ditto	28
Unger's Botanical Letters	29

SURGERY.

Ashton on Rectum	4
Bellingham on Aneurism	5
Brag on Artificial Limbs	6
Bishop on Bones	6
Chapman on Ulcers	9
Do. Varicose Veins	9
Cooper (Sir A.) on Testis	9
Do. (S.) Surg. Dictionary	9
Curling on Rectum	10
Do. on Testis	10
Druitt's Surgery	11
Ferguson's Surgery	11
Fraser on Chest	12
Harrison on Stricture	14
Higginbottom on Nitrate of Silver	15
Hodgson on Prostate	15
James on Hernia	17
Jordan's Clinical Surgery	17
Laurence on Cancer	18
Lawrence on Ruptures	18
Liston's Surgery	18
Macleod's Surgery of the Crises	19
Macilise on Fractures	19
Nunneley on Erysipelas	21
Pemberton on Melanosis	22
Pirrie on Surgery	22
Smith on Stricture	25
Snow on Chloroform	25
Steggall's Surgical Manual	26
Teale on Amputation	27
Thompson on Stricture	27
Do. on Prostate	27
Watson on the Larynx	29
Wilson on the Skin	30
Do. Portraits of Skin Diseases	31
Yearley on Deafness	31
Do. on Throat	31

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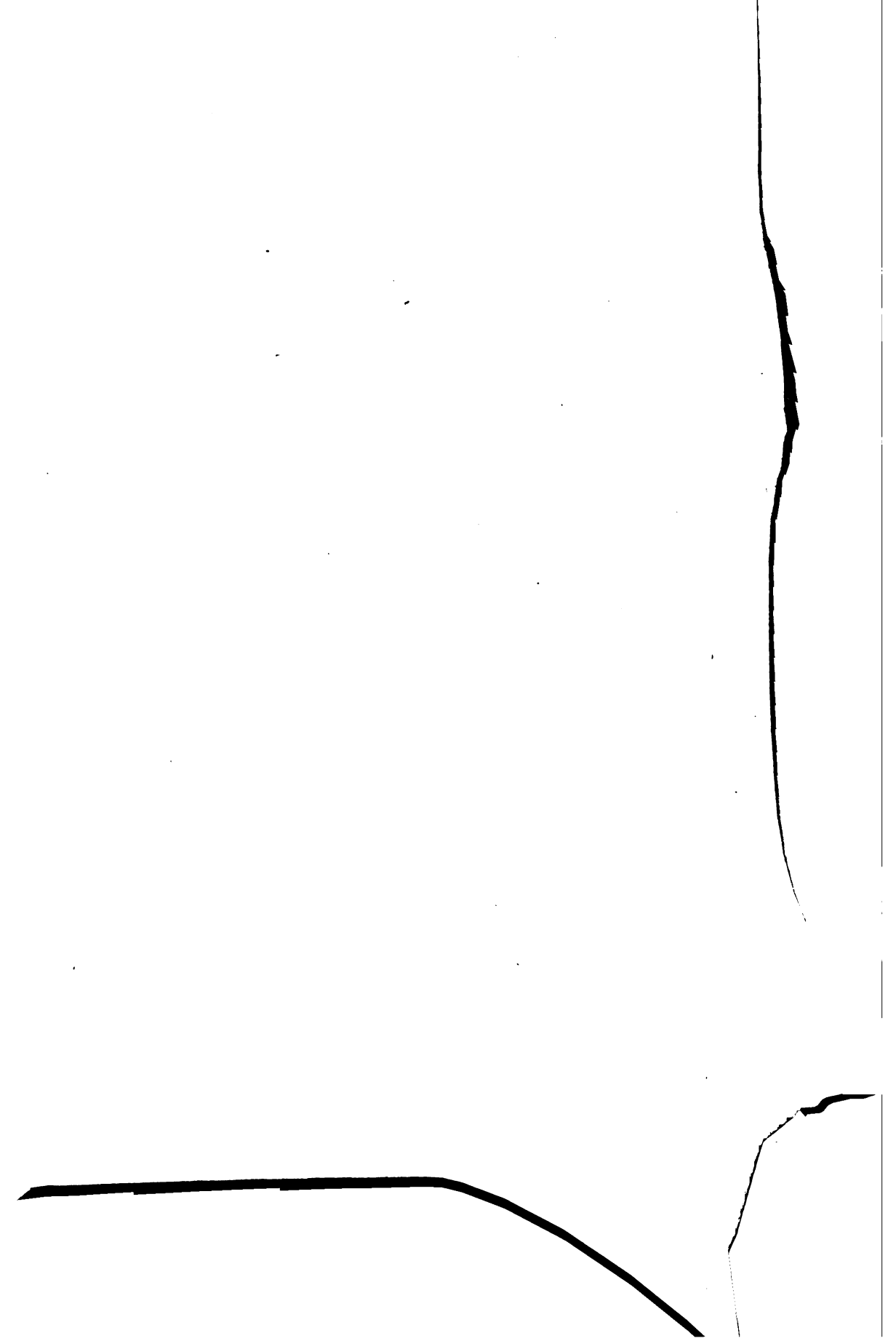
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